

# METALLURGIA

THE BRITISH JOURNAL OF METALS

CASE No. 4.

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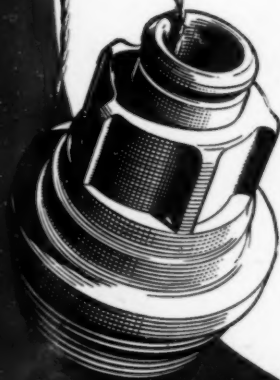
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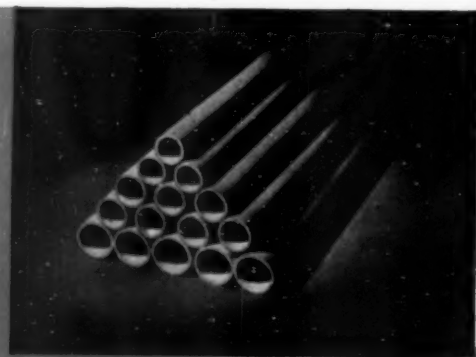
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# METALLURGIA

## *The British Journal of Metals*

(INCORPORATING THE METALLURGICAL ENGINEER)

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### PRINCIPAL CONTENTS IN THIS ISSUE:

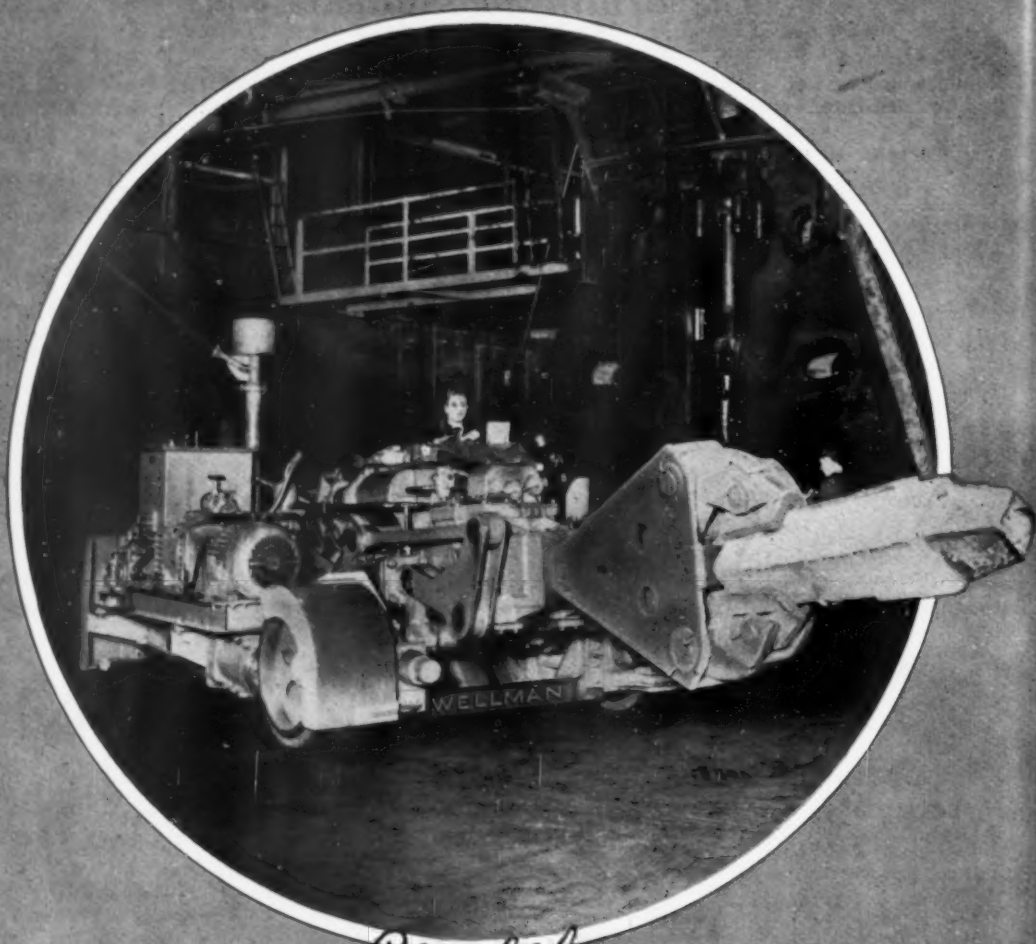
	Page		Page
Industry and Steel .. . . .	69	The Late Mr. David Flather .. . .	94
Investigational and Experimental Laboratories in Australia .. . .	70	The Iron and Steel Institute .. . .	95-103
At the Basle Fair .. . . .	70	<i>The annual general meeting of this Institute is reported, the President's Address is summarised and some of the main aspects are given of papers presented at the technical sessions and of the discussions which followed.</i>	
A Refined Metallographic Technique for the Examination of Surface Contours and Surface Structure of Metals. By A. J. W. Moore, Ph.D.	71-74	Combating Corrosion of Iron and Steel	104-106
<i>A detailed account is given of a taper-sectioning technique, which can be regarded as a refined method for the examination of surface contours and structures of metals.</i>		<i>The scope and practical character of the work of the Corrosion Committee of the British Iron and Steel Research Association and its Sub-Committees.</i>	
The Adhesion of Enamel Finishes to Electro-Plated Cadmium Coatings. By E. E. Halls .. . . .	75-78	The Institution of Metallurgists .. . .	107
<i>Results are summarised of laboratory tests on cadmium-plated steel panels having various types of enamel finish, and on which the cadmium was given various chemical treatments prior to enamelling.</i>		The Institute of British Foundrymen	107
A New Range of Aerofoil Fans .. . .	78	The Chemical Analysis of a Permanent Magnet Alloy. By C. H. R. Gentry	108-113
Some Developments in Steels for Welded Structures. By W. Barr, A.R.T.C., F.I.M. .. . . .	79-84	<i>Procedures are given for the determination of cobalt, nickel, aluminium, copper, titanium, silicon and manganese. A significant feature is the use of photometric and polarographic methods for the determination of elements present in high percentages.</i>	
<i>Certain characteristics of steel have assumed importance where welding is concerned, and these were discussed in this paper.</i>		The Colorimetric Estimation of Tungsten in High Speed Steels Using Ammonium Thiocyanate and Titanous Chloride. By R. St. J. Emery and D. W. Curtis .. . .	113-114
Melting Furnaces in German Steel Foundries. By Dr. Ing. Hans Stein and Dr. Ing. Karl Roesch. .. . .	85-89	Group Separations in Inorganic Qualitative Analysis. Part I. Schemes based on Sulphuretted Hydrogen. By F. R. M. McDonnell and C. L. Wilson .. . . .	115-118
<i>A report on trends in Germany of various types of steel-making furnaces used in steel foundries.</i>		Miscellaneous Microchemical Devices: XIII A Micro-Mortar for Powdering Solids .. . . .	118
Exhibition of Zinc Alloy Die Castings	90-91	XIV A Hydrogen Sulphide Generator for Microanalysis .. . .	118
<i>Brief report of a recent exhibition of die castings held in Birmingham.</i>		Metallurgical Digests .. . . .	118-122
Correspondence:—			
Gas Carburising with Town's Gas	91-94		

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# METALLURGIA

THE BRITISH JOURNAL OF METALS.

INCORPORATING "THE METALLURGICAL ENGINEER."

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## Industry and Steel

**B** RITAIN'S prosperity, and indeed the maintenance of her basic services, requires a high level of production in steel and the industries associated with it. In spite of the great and successful efforts which the whole steel industry is making, not enough steel is being made for all the urgent work required to be done within Britain, much less is production capable of meeting the needs of her markets overseas. This expression of opinion by Mr. Harold Wilson, President of the Board of Trade, in a speech at the recent Cutlers' Feast held in Sheffield, would be accepted by all present. But he went on to say that the steel industry would have been in a better position to-day if its capacity had not been restricted before the war by the fact that other industries and the community as a whole had failed to make the demands on the steel industry they should have done in their own interests. Doubtless there is some truth in this statement, but the main causes of the present heavy demands on the steel industry are due to the almost complete diversion during the war, of supplies to war purposes. Millions of tons of steel which would normally have been applied to peaceful needs were used for war purposes, and industries at home and abroad were unable to obtain new steel products, replacements, or to effect repairs to existing plant unless they were directly concerned with the war effort.

Since the end of the war the steel industry has been called upon to meet steel demands for peace-time purposes which have accumulated through the war years and, although the industry is tackling the production problem with greater success than probably any other industry, it will be several years yet before some degree of balance is reached between the supply and demand of steel; and only then will it be possible to determine what can be regarded as the most economical plant capacity to meet normally expanding demands.

Before the war, many iron and steel firms had carried out extensive reorganisation schemes at considerable cost. Many blast furnaces were rebuilt, steel furnaces and rolling mills were installed or improved and these firms were well equipped to meet the great emergency created by the outbreak of war. Some firms were only in the initial stages of reconstruction when war was declared and were forced to shelve their schemes in view of the emergency. During the war, all steel plants worked as near as possible to their maximum capacity and, in consequence, on its termination not only was considerable new plant needed, but it was necessary to overtake the arrears of repairs and maintenance as quickly as possible, because of the short time lag between the steel demands for war and those for peace. Indeed, this time lag proved to be almost imperceptible and improvements and plant renewals have had to be carried out while still striving to produce

at capacity. That the industry is doing so well, not only in the volume of production but in the quality of its products, is a credit to both managements and men.

A continuing demand and a continuing challenge to the exporting industries were referred to by Mr. Wilson, who emphasised the fight Britain must win to ensure her national survival and her ability to pay her way abroad. In the first four months of this year, he said, the industries depending on steel have been exporting at the annual rate of £580 million, or over 40% of the total national exports. Our exports of steel in direct or indirect form are at present enough to pay for 70% of our food imports, or 87% of our raw material imports. In exports to the Western Hemisphere, which represent a high priority in the national effort, the steel and steel using industries have been exporting in the last four months at the rate of £63 million a year, over one-third of our total efforts, to that area. While these refer to the volume of exports, their value in terms of bargaining power far exceeds the value in statistical terms.

Although exports are a first priority on British industries, the task of re-equipping these industries must be given a second priority. In view of the heavy calls made on industrial plant and machinery during the war a large part of industry needs to be capitalised with new and modern capital equipment. Many basic industries are re-equipping themselves at an encouraging rate and, in this respect, particular mention may be made of the iron and steel industry which has considerably increased its blast furnace and steel furnace capacities since the end of the war, in addition many modifications of plant have been carried out, not only with a view to increasing production but also to effect improvements in the quality of products.

In discussing steel, Mr. Wilson referred particularly to Sheffield and her industries, mentioning Sheffield cutlery, Sheffield plate, and Sheffield steel, names that are associated with quality throughout the world. Sheffield, he said, led world technique in steel making with Huntsman and Bessemer; contributed to an important section of consumer goods by Brearley's discovery of stainless steel; and revolutionised machine shop practice, and with it the engineering industry, with her high speed steel. To-day, Sheffield's reputation as a steel centre is higher than ever, her newest metallurgical developments are associated with gas turbine materials, and in all sections of the steel industry she is continuing to play a leading part.

British industry to-day is faced with a challenge, a challenge of paying her way, which can only be met by increased production, by harder work, and by the fullest play of inventiveness. Britain must regain her strength after the exhaustion of two world war victories. Success will mean the re-establishment of her power and leadership in the world and the steel industry can be relied upon to give the maximum assistance to achieve it.

## Investigational and Experimental Laboratories in Australia

MANY overlook the great industrial progress in Australia in recent years and the considerable developments being made in the equipment of laboratories for investigational work, which, previous to the recent war, was carried out on a very limited scale. During and since the war, however, the position has changed to a remarkable extent, largely as a result of the development of the Munitions Supply Laboratories originally established for the testing of munitions and related products. Since 1940, the work of these Laboratories has been greatly extended and investigational work of a defence character was undertaken, on somewhat similar lines to that undertaken at Woolwich Laboratories in this country. When the war ceased the chemical, physical, metallurgical and engineering sections of the Laboratories were probably better equipped than any other in Australia.

Since very few private firms in Australia have facilities for carrying out investigational work, it was decided to permit the Munitions Supply Laboratories to carry out work for private firms, when facilities in other laboratories were not available. This policy has proved to be very popular and some indication of its success will be noted from the fact that approximately half the work now being carried out at these Laboratories is on behalf of private firms. In general, the work is of an investigational character and is primarily concerned with improved production methods, particular attention being directed to the determination of causes of production rejects, and is charged for on a materials and labour costs basis.

Of considerable interest is an experimental foundry section, established primarily to apply overseas developments to Australian conditions, and also to assist in the production of castings for munitions or aircraft. Some investigational work for private foundries has already been carried out, and considerable work is in progress at the present time. This foundry is the only true experimental foundry in Australia, and it seems apparent that it is supplying a long felt need. It is planned to finally equip this foundry with all the major melting equipment including an 80 kw. lift-out coil induction melting unit, a 150 lb. capacity oil fired crucible furnace, several gas-fired crucible furnaces, and a 25 lb. capacity arc furnace. A complete range of pyrometric and gas analysis equipment will also be installed, as well as a sand testing laboratory and sand mixing and casting cleaning sections.

The present programme of work in this experimental foundry includes the development of centrifugal casting, very little of which has been done in Australia; cement moulding and precision casting, and a study of a foundry type instrument for the determination of gas contents of molten metals and alloys, which has been received from Germany as a part of reparations. This German "Straube-Pfeiffer" equipment is somewhat similar in type to that developed by the British Non-Ferrous Metals Research Association, but it is believed to be cheaper to make and, as it works at a pressure of  $\frac{1}{2}$  to 2 mm. compared to a pressure of 30 to 40 mm. of the Association's apparatus, it is claimed to be capable of giving more accurate estimates of low gas contents.

The fact that the Munitions Supply Laboratories have a staff of approximately 900, with branches in Sydney and Adelaide, indicates that research and development work in Australia is progressing at a rapid rate.

## At the Basle Fair

QUITE the most valuable new product at this year's Swiss Trade Fair, for the metal industry at least, was Camille Bauer's new total radiation pyrometer. The essential part of this pyrometer is the delicate thermopile detector containing up to 36 very fine thermocouples in series and the success of the instrument is almost entirely due to the fine manufacturing limits of this near microscopic detecting element.



An aerial view of the Basle Fair.

There are three ranges of instrument: 800-2,000° C., 500-1,500° C. and 100-1,000° C. and their 16, 24 and 36 unit elements give 159, 94 and 37 m.v. respectively at full scale reading across internal resistances of 50, 70 and 100 ohms to deflect suitable voltmeters or galvanometer systems. The very fine detector system gives its advantages as high accuracy, better than 5° C. is quoted at 1,500° C., and to take full advantage of this a sensitive milli-voltmeter should be used.

This instrument is primarily designed as a permanent fixture on furnaces, but with a slight adaptation it is clearly a valuable instrument for determining pouring and casting temperatures of flowing molten metals.

An outstanding novelty was the widespread use of glass-wool as an insulator in all types of electric motors. This should greatly reduce the fire risk in foundries and machine shops. The Swiss are naturally well advanced in making and designing artificial jewel bearings and mountings due to the natural demand of their highly developed fine instrument and watch industries, and these were a speciality displayed by many exhibitors at the Fair.

In such a competitive atmosphere the display, design and advertising standards were much higher than in England with a general aptitude for ingenuity, attractiveness and eye-catchiness. Every machine which moved, rotated, cut or shaped was working, shaping metal on lathes, cutting gears and pressing parts.

Perhaps typical of the *en fête* spirit of the whole town were the advertising trolleys towed behind the trams in which you left the Fair and the huge captive balloon on high advertising a well known patent medicine. One can hardly imagine similar trucks, advertising X's stainless steel and Y's diamond dies following you as you left the B.I.F. at Birmingham, but that is merely the difference between British and continental showmanship.

# A Refined Metallographic Technique for the Examination of Surface Contours and Surface Structure of Metals

## Taper Sectioning

By A. J. W. Moore, Ph.D.

*Tribophysics Section, C.S.I.R., Australia, and Research Group on the Physics and Chemistry of Rubbing Solids, Cambridge*

*A detailed account is given of taper-sectioning technique as a means of studying the surface features of metals. The method is not new but it has not been applied to any great extent, although in many respects its use permits closer study than the normal cross-section micro-examination method. It can be regarded as a more refined method for the examination of surface contours and structures of metals and has many useful applications.*

A STANDARD method of studying surface irregularities is to cut a cross-section for micro-examination. Since there is no distortion ratio between the horizontal and vertical component of the contours, the highest powers must be used to observe the finer irregularities. The area of field is, therefore, very much restricted. Also the minimum size of irregularity observable is limited by the resolution of the microscope. Provided that the surface irregularities are elongated in one direction—i.e., scratches caused by grinding, score marks produced by friction and wear, etc., either of these limitations can be overcome to a considerable extent by cutting the section obliquely to the surface, thus obtaining a taper section with the vertical component of the surface contour greatly magnified, relative to the horizontal one. Fig. 1 shows diagrammatically the effect of cutting a section at an angle  $\alpha$  to a surface containing a V-shaped groove. The distortion ratio (i.e., the ratio of the magnification in the vertical and horizontal directions in the final section) is the cosecant of the angle. Thus a section cut at an angle of  $5^\circ 43$  mins. would give a distortion ratio of ten.

A typical taper section is shown in Fig. 2. It is a scratch of a steel pin in a copper surface and shows the high built-up edge, and the area of work-hardened copper beneath the scratch. As will be seen later, the distortion ratio of each taper section can be measured to within 5%, and this ratio is incorporated in the magnification diagram at the top of the figure.

Figs. 3 and 4 show taper sections of steel surfaces which have been finished with 150 and 600 grade wet carborundum polishing papers respectively. The finer carborundum gives an almost mirror-like surface which is shown on sectioning to have measurable scratches. It is proposed to give here a full account of the technique used in the hope that others may find it useful in any studies of the surface features of metals. The idea of cutting a section obliquely to the surface is of course not new, and many people have no doubt used it. However,

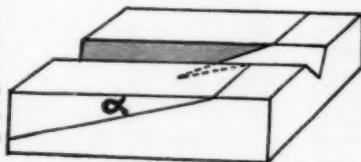


Fig. 1.—Diagram showing the magnification caused by cutting a taper section at an angle.

it appears to have first been described by Nelson<sup>1</sup> (1940). Essentially, his method consists of first grinding the bottom face of the specimen parallel to the one to be examined which he then protects with a heavy electrodeposit of nickel. The specimen is then mounted on an accurately made wedge and ground down by a surface grinder until the ground surface extends across the whole upper face and the steel nickel interface cuts across the specimen near its centre.

The modification of his technique which is used for this work allows one to make a taper section of any clean metal surface and also to measure the angle of sectioning so that the vertical magnification may be accurately known. In addition, the metallographic polishing of the section is simplified since the specimen is mounted in a plastic mount of convenient size for holding on the polishing cloth. The angle at which the section is to be taken is determined by the shape of the follower in the

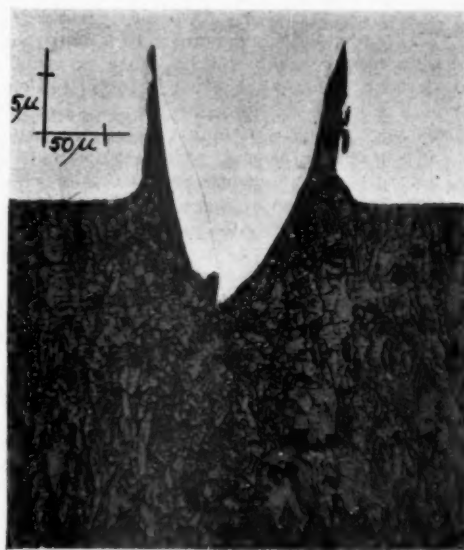


Fig. 2.—Taper section of a scratch drawn on copper with a steel point. Note the high built-up edge and the deformation below the surface.

<sup>1</sup> Nelson, H. R., 1940, Conf. on Friction and Surface Finish, M.I.T., p. 217.



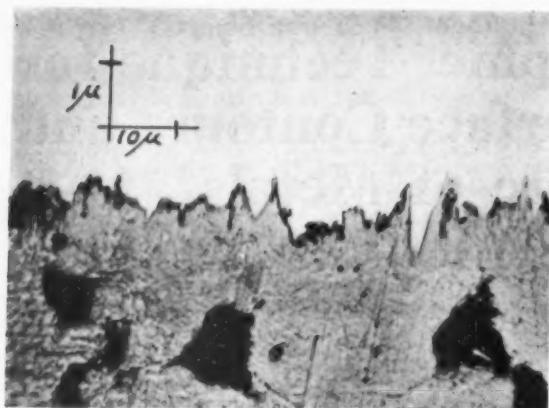


Fig. 3.—Taper section of the contours of a mild-steel surface produced by rubbing with a 150 grade wet carborundum paper.

mould of a standard bakelite press used for mounting metallographic specimens. Several sections may be made from one specimen and their positions accurately determined. The method is best described by means of a series of diagrams (Fig. 5).

The specimen is represented by I. The front surface, which is the one to be examined, is marked A. Its size is limited only by the dimensions of the mould in the press. In order that the specimen will key into the plastic its sides must be sloped back as in II. This can be done on all four sides with a small grinding wheel, and it is essential that the edges at *c* be sharp. The back of the specimen is rough ground and filed so that it is reasonably flat and approximately parallel to the front surface.

The next stage is the protection of the front surface by the electro-deposition of a metal of similar hardness to the specimen. Most of the sections made by the author have been of steel or copper, and it has been found that a nickel deposit is quite suitable for protecting steel, while silver is quite satisfactory for copper surfaces. Although the procedure described here is especially suitable for steel or copper surfaces it is quite possible to make satisfactory sections of other metals if a supporting layer of suitable hardness can be provided which will adhere very strongly without necessitating deep etching of the surface and so destroying its original contour.

The degreasing procedure prior to electroplating must be such that the contours are not altered by it (i.e., the very effective but drastic method of rubbing with an abrasive must not be used). It was found that cathodic cleaning in a hot alkaline bath at a current density above 5 amp./dm<sup>2</sup> gave a clean surface. A suitable composition would be 10% anhy. sodium carbonate and 2% sodium hydroxide in water and used at about 80°–90° C. The anode is a loop of platinum wire and the specimen is made the cathode. The high rate of hydrogen evolution from the surface of the specimen is an effective mechanical aid to the detergent properties of the alkali and after about 20 seconds the surface should be grease free. It is next thoroughly rinsed in running water and inspected for cleanliness. The water must wet the specimen completely. If not, then it must be returned to the cleaning bath.

The etching of the surface prior to plating should be reduced to the barest minimum. Nelson (*loc. cit.*)

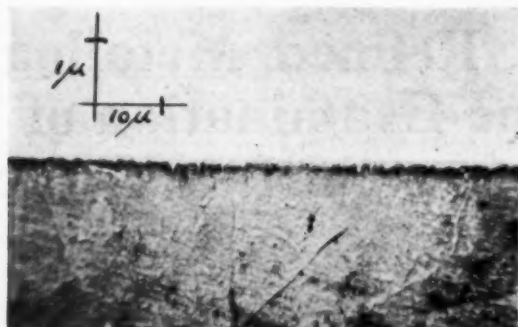


Fig. 4.—Taper section of the contours of a mild-steel surface produced by rubbing with a 600 grade wet carborundum paper.

states that a 5–10 secs. dip in 0.1 N HCl produces no detectable change in contour and the very light etch so produced will give the nickel the necessary adherence. Also, the acid removes the last traces of alkali from the surface so preventing precipitation in the nickel bath and bad adhesion of the deposit. Nickel plating is carried out in a standard Watts type bath.

#### Composition:

NiSO <sub>4</sub> · 6H <sub>2</sub> O	250 gm.
KCl	21 gm.
H <sub>3</sub> BO <sub>3</sub>	29 gm.
Water	1 litre

at a temperature of 40°–50° C. and current density 3 amp./dm<sup>2</sup>. The buffering action of the boric acid is necessary in order to keep the pH between 3.0–3.5. If the bath is not initially between these limits then it must be adjusted either with H<sub>2</sub>SO<sub>4</sub> or KOH. Deposition should continue for about 7½ hours, giving about 0.010 in. of nickel.

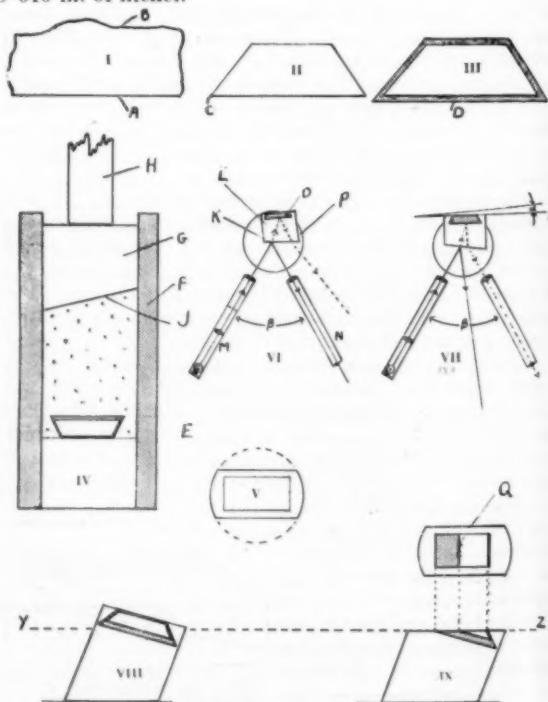


Fig. 5.—The technique of taper sectioning.



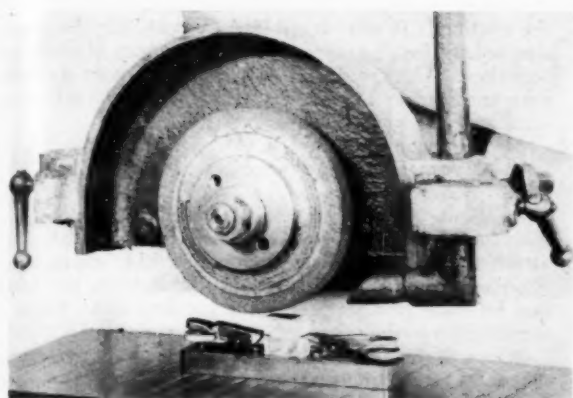


Fig. 6.—The process of grinding a taper section.

The silver deposit on copper is applied from a standard double cyanide bath. Again it is essential that the specimen be completely grease free but no etching of the copper prior to plating is necessary.

The composition is

AgCN .. .. .	26-27 gm.
KCN .. .. .	45-48 gm.
K <sub>2</sub> CO <sub>3</sub> .. .. .	35-40 gm.
Water .. .. .	1 litre

The bath is used cold at a current density of 0.4 amp./dm<sup>2</sup>. In order to obtain good adhesion it is essential that the current is flowing immediately the copper is put in the bath. At this current density 0.010 in. will be deposited in about 20 hours. The specimen after plating (III) must be polished to a mirror-like finish on the surface of the electrodeposit.

The specimen is now ready for mounting in plastic; IV shows a cross-section of the mould of the press. The anvil E closely fits into the Tube F. The specimen and moulding powder are placed in this tube and the follower G inserted. Pressure is applied by means of the plunger H and the whole is heated by a pair of heating tongs which clamp around the tube F. The angle of the follower is the same as the final angle of the section. In all the photomicrographs shown here, this was

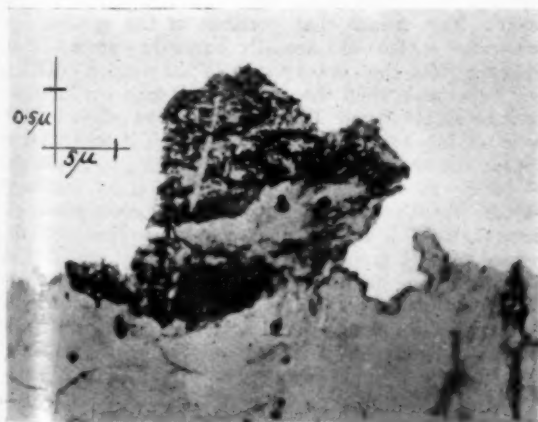


Fig. 8.—Taper section of copper particle which has seized on to a steel surface after sliding. Note how the steel has been pulled away by the copper.

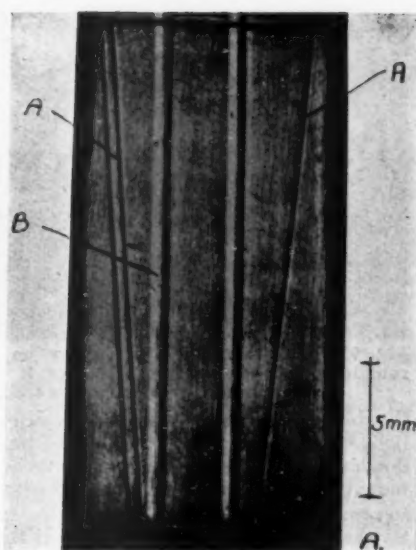


Fig. 7.—A method of locating a taper section by diverging scratches.

5° 43 mins. to give a distortion ratio of 10 : 1. Since the angle will be accurately measured later, it is not essential that the follower be made very accurately.

The specimen is inserted in the tube with the back face against the anvil and enough powder added to give about 1/4 in. of plastic above the specimen in the finished moulding. A transparent plastic must be used and the thermoplastic methyl-methacrylate type (e.g., Diakon ex I.C.I.) is quite suitable. The sloping face of the follower should be highly polished so as to impart a good finish to the plastic. If the surface irregularities which are to be examined tend to be elongated in one direction, then the slope of the follower must be in the same direction as these irregularities.

Upon removal from the press the plastic is cut away to give two parallel flat faces perpendicular to the surface of the nickel plating. This gives a plan view as in V. Since the back of the original specimen was only made approximately parallel to the front face and also since the follower may slip a little sideways when moulding, the angle X between the face K and the plated surface D will vary slightly, but this is not important if it can be measured accurately.

This can be done in several ways. One of these which was best suited to the apparatus available is as follows:

The specimen (L) is put on its side on the table (P) of a goniometer which is provided with a light source and slit in one tube (M) and a telescope in the other (N). (See VI). The tubes are set at a definite fixed angle, during



Fig. 9.—Taper section of a groove on copper after a rounded steel contact has slid over it once. Note the deformation below the surface.

the determination, and the table is adjusted so that the light from the slit is reflected by the plastic surface (K) down the telescope. The angle through which the table must be turned so that the refracted ray passes down the telescope is then accurately measured (see VI and VII for the two positions of the table). If the angle between the two positions is  $\alpha$  and the angle between the tubes  $\beta$  then:

$$X = \frac{1}{2} \arcsin \frac{\sin(\frac{\beta}{2} + \alpha)}{\mu} - \arcsin \frac{\sin(\frac{\beta}{2} - \alpha)}{\mu}$$

The relative magnification in the taper section is cosecant  $X$ . It is possible to draw a graph relating cosec  $X$  with  $\beta$  at some fixed value (say  $60^\circ$ ) and  $\mu = 1.495$  which is the value for methyl-methacrylate.

The specimen is now ready for grinding on a surface grinder. This is a familiar machine tool in which a table with the specimen clamped to it is passed back and forth beneath an abrasive wheel turning at high speed. The table is slowly raised and the wheel takes successive fine cuts right across the specimen which has been inverted so that the sloping face K is next to the table of the grinder. Grinding continues until the interface between the nickel and the steel is exposed. A cooling fluid must be used. This is shown diagrammatically in VIII. If it is ground down to the line YZ, the plan view of the specimen will be similar to that in IX and the taper section is shown at Q. Fig. 6 shows the specimen mounted on the magnetic table beneath the grinding wheel. Two small saw cuts in the plastic enable it to be rigidly clamped to a steel jig. Subsequent polishing and etching of the specimen was carried out by standard methods (e.g., Kehl<sup>2</sup>, 1943). Some difficulty was experienced in eliminating all relief between the nickel and steel, but it was found that grinding in wet carborundum papers followed by the minimum of polishing with alundum on a Selvyt wheel gives the best results. After polishing, a good contrast is obtained with nickel and steel by etching for about 5 seconds in 2% nitric acid in ethyl alcohol. In this way the steel only is etched and its structure is revealed.

It was found much more difficult to obtain a suitable etchant in the case of a copper specimen protected with silver plating. The flowed layer of copper and silver caused by the polishing is much thicker than with steel and when the copper is etched sufficiently to reveal the underlying crystal structure, there is frequently found a distinct step between the copper and the unetched silver. This shows up as a "ghost" band running parallel to

the contour. It can be reduced by polishing the specimen lightly, leaving some of the more persistent scratches and then etching as little as possible. Many etchants were tried out and the best appeared to be the following acid ferric chloride mixture:

Ferric chloride .. .. .	10 g.
Hydrochloric acid (conc.) .. .	30 c.c.
Water .. .. .	120 c.c.

with about 30–50% conc. HCl added just before use. Also the effect of this dark band can often be reduced by altering the lighting conditions of the microscope. Frequently, when using vertical illumination the band can be made to disappear if the incident light makes a small angle to the optic axis.

When the contours of a particular scratch or groove are being studied it is often necessary to know the place where the taper section has been cut. Diverging deep scratches A are ruled on the surface before plating, one on each side of the groove B and the whole surface photographed at a known magnification. If two tracks are to be sectioned on the same specimen, then two scratches may be drawn on one side in order to distinguish between the tracks (Fig. 7A). When the taper section is cut its exact position can be determined by measuring the distance between these scratches in the section (CD of Fig. 7B) and comparing this with the photograph.

This technique of taper sectioning has been found very useful by the author for studying the mechanism of surface damage due to sliding metals. It has been shown, for instance, that the particles of copper which have been "picked up" on a steel surface after a loaded copper slider has passed over it at slow speed and which are too small to be observed by normal methods, can be studied in detail in taper sections. Thus, Fig. 8 shows how a copper particle can pluck steel out of the surface. Also, the metal immediately below the surface can be examined. Taper sections of the track formed when a rounded steel slider slides on copper, show considerable sub-surface deformation (Fig. 9). Without the relatively greater vertical magnification of the taper section this zone of deformation would have been too narrow to detect readily. These experiments, and examples of other taper sections, are described in greater detail by Bowden and Moore<sup>3</sup> (1947).

It should always be remembered in interpreting taper sections, that they have been cut out at an oblique angle. This means that portions of the section seen below the surface are actually below the surface some distance from the contour seen in the section. This is not important when the contours of long irregularities such as scratches are being studied, but when a lump or a pit has been taper sectioned then it will tend to appear too small. Thus, any measurements of the size of lumps or pits should be considered as minimum values. However, if this limitation is borne in mind, it is nevertheless clear that for many applications this refined metallographic method is a very useful tool. This technique has been used by the author (an officer of Tribophysics Section, C.S.I.R., Australia) at the Research Group on the Physics and Chemistry of Rubbing Solids, Department of Physical Chemistry, Cambridge.

Thanks are due to Dr. F. P. Bowden for his constant help and encouragement and to the Council for Scientific and Industrial Research (Aust.) for making this work possible.

<sup>2</sup> Kehl, G. L. 1943, *The Principles of Metallographic Laboratory Practice*, McGraw-Hill, New York.

<sup>3</sup> Bowden, F. P., and Moore, A. J. W., 1947, *Inst. of Metals Symposium on Internal Stresses in Metals and Alloys*, p. 131–137.

# The Adhesion of Enamel Finishes to Electro-Plated Cadmium Coatings

By E. E. Halls

*Extensive laboratory tests have been carried out on cadmium-plated steel panels having various types of enamel finish and on which the cadmium was given various chemical treatments prior to enamelling. The results are summarised in tabular form*

**T**HERE are extensive applications for electro-plated coatings, particularly on iron and steel but often on non-ferrous metals, with the subsequent application of organic finishes of enamel, paint or lacquer.

When the electro-plate coating consists of cadmium difficulties are always encountered with enamel adhesion. These difficulties are even more marked than with other electro-plate coatings, such as zinc, copper or nickel and, in fact, it is usually stated that cadmium coatings, without some chemical pre-treatment, cannot be satisfactorily enamelled. The evidence from Laboratory tests and experience in the field is that this statement is correct.

The following summarises briefly some fairly extensive laboratory tests on cadmium plated steel panels having various types of enamel finish and on which the cadmium was given various chemical treatments prior to the enamelling. The benefit of these pre-treatments was assessed from the condition of the enamel, with particular reference to brittleness and adhesion in the initial condition after enamelling, as well as after extended exposure to humidity and atmospheric tests. The humidity test comprised exposure of the panels to a number of daily cycles in which the temperature for the 8-hour day was 55–60° C. with a humidity of 65–75%, and the night temperature was that obtained by cooling down to that of the atmosphere with a rise in the humidity to 100%, and with moisture condensation upon the specimens.

The atmospheric test merely comprised open exposure

without protection from direct rain, etc., on a frame facing south-west in a semi-industrial atmosphere. The quality of the enamel at any stage was assessed by scratch and bend tests. The scratch test was made using a hardened steel tool, one end of which was tapered to the frustum of a cone, conical angle 15° and diameter of frustum  $\frac{1}{16}$  in. With this tool a number of scratches were made at oblique angles so as to cross one another and adhesion was evaluated by the degree of chipping or flaking that occurred, and the ease with which the enamel could be removed from the angle between two such scratches.

The bend test merely comprised bending slowly through 180° with the finished side of the panel outwards and the radius of the bend approximately  $\frac{1}{2}$  in.

The best results obtained are briefly tabulated on a basis of the type of enamel finish.

Table I deals with pigmented cellulose enamel finish. The data are self-explanatory and initially it is seen that the only useful finish is represented by Sample No. 4 with phosphate passivation No. 2. However, none of the finishes show any real permanency and it is concluded that ordinary cellulose enamel finishes are unsatisfactory, on a cadmium plate base.

Table II also deals with a cellulose finish, in which the first coating is a clear lacquer of rather special composition, because it is a combination of nitro-cellulose and glyptal-resin medium. On the initial assessment, Samples No. 3 and 4, having phosphate passivation No. 1 and No. 2 respectively, are both good. They show this superiority throughout the test, although only the

TABLE I.—AGEING TESTS ON ENAMEL FINISHES UNDER DURABILITY TESTS. CADMIUM-PLATED STEEL PANELS WITH AND WITHOUT PASSIVATION, ENAMELLED WITH TWO COATS OF PIGMENTED CELLULOSE ENAMEL

Sample No.	Chemical Treatment of Cadmium Plate	Exposure Conditions	Condition of Enamel Finish Judged by :		Assessment of Enamel Finish
			Scratch Test	Bend Test	
1 .. .. .	None.	None—initial condition of enamel finish.	Brittle, chipping badly.	Cracks and flakes very easily.	Very poor.
2 .. .. .	Chromate Passivation.	ditto	Rather brittle, chips.	Cracks and removed easily.	Poor.
3 .. .. .	Phosphate Passivation No. 1	ditto	A little brittle, chipping slightly.	No cracking or flaking, very good.	Good.
4 .. .. .	Phosphate Passivation No. 2	ditto	Clean cut, no chipping, very good.	No cracking or flaking, very good.	Excellent.
1 .. .. .	None.	Humidity—7 days.	Chipping and flaking very badly.	Cracking and flaking completely away.	Exceedingly poor.
2 .. .. .	Chromate Passivation.	" 30 "	ditto	ditto	ditto
3 .. .. .	Phosphate Passivation No. 1	" 7 "	Chips rather badly.	ditto	ditto
4 .. .. .	Phosphate Passivation No. 2	" 30 "	Chips very badly.	ditto	ditto
		" 7 "	Chips badly.	ditto	ditto
		" 30 "	Clean cut, no chipping, very good.	Cracking and flaking easily.	Fair.
		" 7 "	Very slight chipping, still quite good.	ditto	Fair.
1 .. .. .	None.	Open atmosphere—30 days	Chipping and flaking very badly.	Cracking and flaking completely away.	Exceedingly poor.
2 .. .. .	Chromate Passivation.	ditto	Chipping and flaking.	Cracking and flaking completely away.	Very poor.
3 .. .. .	Phosphate Passivation No. 1	ditto	ditto	ditto	ditto
4 .. .. .	Phosphate Passivation No. 2	ditto	ditto	Cracking and flaking easily.	ditto



TABLE II.—AGEING TESTS ON ENAMEL FINISHES UNDER DURABILITY TESTS. CADMIUM-PLATED STEEL PANELS, WITH AND WITHOUT PASSIVATION, ENAMELLED WITH ONE COAT OF SPECIAL CLEAR CELLULOSE LACQUER AND ONE COAT OF PIGMENTED CELLULOSE ENAMEL

Sample No.	Chemical Treatment of Cadmium Plate	Exposure Conditions	Condition of Enamel Finish Judged by :		Assessment of Enamel Finish
			Scratch Test	Bend Test	
1 . . . . .	None.	None-Initial condition of enamel finish.	Satisfactory, clean scratch, no chipping or flaking.	Finish cracks, but does not flake and adhesion is good.	Very good.
2 . . . . .	Chromate Passivation.	ditto	A little chipping, no flaking, condition quite good.	Cracks and flakes away.	Poor.
3 . . . . .	Phosphate Passivation No. 1	ditto	No chipping or flaking.	No cracking or peeling.	Excellent.
4 . . . . .	Phosphate Passivation No. 2	ditto	ditto	Finish cracks, but adheres well.	Very good.
1 . . . . .	None.	Humidity—7 days	Bad chipping and flaking.	Finish cracks and flakes clean away.	Exceedingly poor.
2 . . . . .	Chromate Passivation	" 30 "	ditto	ditto	ditto
		" 7 "	Chips, but does not flake easily.	ditto	Very poor.
3 . . . . .	Phosphate Passivation No. 1	" 30 "	Bad chipping and flaking.	Cracks, and finish fairly easily removed.	Exceedingly poor.
		" 7 "	Very slight chipping.	ditto	Moderate.
4 . . . . .	Phosphate Passivation No. 2	" 30 "	No further deterioration.	Satisfactory, no cracking or flaking.	Moderate.
		" 7 "	Satisfactory, no chipping or flaking.	Cracking, but flaked off only with difficulty.	Excellent.
		" 30 "	Very slight chipping, no flaking.	ditto	Very good.
1 . . . . .	None.	Open atmosphere—30 days	Slight tendency to chip.	Satisfactory, no cracking or flaking.	Very good.
2 . . . . .	Chromate Passivation.	ditto	Bad chipping and tendency to flake.	Cracking and flaking away easily.	Exceedingly poor.
3 . . . . .	Phosphate Passivation No. 1	ditto	Satisfactory, no chipping or flaking.	Satisfactory, no cracking or peeling.	Excellent.
4 . . . . .	Phosphate Passivation No. 2	ditto	Satisfactory, no chipping or flaking.	ditto	Excellent.

TABLE III.—AGEING TESTS ON ENAMEL FINISHES UNDER DURABILITY TESTS. CADMIUM-PLATED STEEL PANELS, WITH AND WITHOUT PASSIVATION, ENAMELLED WITH ONE COAT OF PIGMENTED STOVING ENAMEL

Sample No.	Chemical Treatment of Cadmium Plate	Exposure Conditions	Condition of Enamel Finish Judged by :		Assessment of Enamel Finish
			Scratch Test	Bend Test	
1 . . . . .	None.	None-Initial condition of enamel finish.	Some chipping and flaking.	Cracking and flaking easily.	Rather poor.
2 . . . . .	Chromate Passivation.	ditto	Satisfactory, no chipping or flaking.	Satisfactory, no cracking or peeling.	Excellent.
3 . . . . .	Phosphate Passivation No. 1	ditto	ditto	ditto	Excellent.
4 . . . . .	Phosphate Passivation No. 2	ditto	ditto	ditto	Excellent.
1 . . . . .	None.	Humidity—7 days	Chipping and flaking.	Cracking and flaking clean away.	Very poor.
2 . . . . .	Chromate Passivation.	" 30 "	Chipping and flaking badly.	ditto	Exceedingly poor.
		" 7 "	Satisfactory, no chipping or flaking.	Cracks and peels off easily.	Excellent.
3 . . . . .	Phosphate Passivation No. 1	" 30 "	Bad chipping and flaking.	Cracks and fairly easily flaked off.	Exceedingly poor.
		" 7 "	Chips rather badly.	Cracks and fairly easily flaked off.	Moderate.
4 . . . . .	Phosphate Passivation No. 2	" 30 "	Chips and flakes badly.	Cracks and peels very easily.	Exceedingly poor.
		" 7 "	Satisfactory, no chipping or flaking.	Satisfactory, no cracking or peeling.	Excellent.
		" 30 "	ditto	Cracks, no flaking, and only scraped off with difficulty.	Very good.
1 . . . . .	None.	Open atmosphere—30 days	Chips and flakes badly.	Cracks and flakes clean away.	Exceedingly poor.
2 . . . . .	Chromate Passivation.	ditto	Chips badly and flakes.	Cracks, and fairly easily flaked off.	Very poor.
3 . . . . .	Phosphate Passivation No. 1	ditto	Chips badly.	ditto	Very poor.
4 . . . . .	Phosphate Passivation No. 2	ditto	Satisfactory, no chipping or flaking.	Satisfactory, no cracking or flaking.	Excellent.

TABLE IV.—AGEING TESTS ON ENAMEL FINISHES UNDER DURABILITY TESTS. CADMIUM PLATED STEEL PANELS, WITH AND WITHOUT PASSIVATION, ENAMELLED WITH ONE COAT OF STOVED ZINC CHROMATE PRIMER AND ONE COAT OF STOVED PIGMENTED ENAMEL

Sample No.	Chemical Treatment of Cadmium Plate	Exposure Conditions	Condition of Enamel Finish Judged by :		Assessment of Enamel Finish
			Scratch Test	Bend Test	
1 . . . . .	None.	None-Initial condition of enamel finish.	Chips and flakes badly.	Cracks and very easily flaked off.	Very poor.
2 . . . . .	Chromate Passivation.	ditto	Chips and flakes badly.	Cracks and fairly easily flaked off.	Poor.
3 . . . . .	Phosphate Passivation No. 1	ditto	ditto	ditto	Poor.
4 . . . . .	Phosphate Passivation No. 2	ditto	Satisfactory, no chipping or flaking.	Satisfactory, no cracking or flaking.	Excellent.
1 . . . . .	None.	Humidity—7 days	Chips and flakes very badly.	Cracks and flakes clean away.	Exceedingly poor.
2 . . . . .	Chromate Passivation	" 30 "	ditto	ditto	Exceedingly poor.
		" 7 "	ditto	ditto	ditto
3 . . . . .	Phosphate Passivation No. 1	" 30 "	ditto	ditto	ditto
		" 7 "	ditto	ditto	ditto
4 . . . . .	Phosphate Passivation No. 2	" 30 "	Satisfactory, no chipping or flaking.	Cracking, but enamel flaked only with difficulty.	Very good.
		" 7 "	ditto	Cracked and enamel easily flaked off.	Moderate.
1 . . . . .	None.	Open atmosphere—30 days.	Chips and flakes badly.	Cracks and flakes clean away.	Exceedingly poor.
2 . . . . .	Chromate Passivation.	ditto	Chips and flakes badly.	Cracks and enamel flakes clean away.	Exceedingly poor.
3 . . . . .	Phosphate Passivation No. 1	ditto	Chips and flakes.	ditto	ditto
4 . . . . .	Phosphate Passivation No. 2	ditto	Satisfactory, no cracking or flaking.	Cracking, but enamel removed only with difficulty.	Very good.



TABLE V.—AGING TESTS ON ENAMEL FINISHES UNDER DURABILITY TESTS. CADMIUM PLATED STEEL PANELS, WITH AND WITHOUT PASSIVATION, ENAMELLED WITH ONE COAT OF RED OXIDE PRIMER AND ONE COAT OF STOVE PIGMENTED ENAMEL.

Sample No.	Chemical Treatment of Cadmium Plate	Exposure Conditions	Condition of Enamel Finish Judged by:		Assessment of Enamel Finish
			Scratch Test	Read Test	
1	None.	None—initial condition of enamel finish.	Slight chipping and flaking.	Cracking and flaking.	Very poor.
2	Chromate Passivation.	ditto	Satisfactory, no chipping or flaking.	Satisfactory, no cracking or flaking.	Excellent.
3	Phosphate Passivation No. 1	ditto	Slight chipping.	Cracking and flaking clean away.	Exceedingly poor.
4	Phosphate Passivation No. 2	ditto	Satisfactory, no chipping or flaking.	Satisfactory, no cracking or flaking.	Excellent.
1	None.	Humidity—7 days	Chipping and flaking badly.	Cracking and flaking clean away	Exceedingly poor.
2	Chromate Passivation.	" 30 "	ditto	ditto	ditto
		" 7 "	Slight chipping.	Cracking, but flaking only with difficulty.	Good.
		" 30 "	Slight chipping and flaking.	Cracking and flaking clean away.	Very Poor.
3	Phosphate Passivation No. 1	" 7 "	Satisfactory, no chipping or flaking.	Satisfactory, no cracking or flaking.	Excellent.
		" 30 "	Slight chipping or flaking.	Cracking and flaking clean away.	Exceedingly poor.
4	Phosphate Passivation No. 2	" 7 "	Satisfactory, no chipping or flaking.	Cracks, but adhesion very good.	Very good.
		" 30 "	ditto	Cracks, adhesion good, flaked off only with difficulty.	Good.
1	None.	Open Atmosphere—30 days	Chipping and flaking.	Cracking and flaking easy.	Very poor.
2	Chromate Passivation.	ditto	Satisfactory, no chipping or flaking.	Cracks, but enamel removed with difficulty.	Very good.
3	Phosphate Passivation No. 1	ditto	Slight chipping and flaking.	Cracks and easily flaked.	Poor.
4	Phosphate Passivation No. 2	ditto	Satisfactory, no chipping or flaking.	Cracks, but enamel removed only with difficulty.	Very good.

TABLE VI.—EXTENDED TESTS ON ENAMEL FINISHED PANELS SPECIMENS No. 4 FROM TABLES II TO V INCLUSIVE (CHEMICAL TREATMENT PHOSPHATE No. 2).

Finish	Further Deterioration When the Test Conditions Were Extended to 6 Months.	
	Humidity Test	Atmospheric Test
Special clear cellulose first coat, pigmented cellulose enamel top coat.	Become very poor.	Become very poor.
Stoved pigmented enamel, one coat.	Very little deterioration	No deterioration.
Stoved zinc chromate primer, stoved pigmented enamel finishing coat.	Very slight deterioration.	Exceedingly slight deterioration.
Stoved Red Oxide Primer, stoved pigmented enamel finishing coat.	No deterioration.	No deterioration.

TABLE VII.—CHARACTERISTICS OF ENAMEL MEDIA USED FOR TESTS IN TABLES No. 1 to 6.

	Pigmented Cellulose Enamel.	Clear Cellulose Lacquer (Special Type)	Stoving Pigmented Enamel.	Stoving Zinc Chromate Primer.	Stoving Red Oxide Primer.
Type	Heavy bodied grey pigmented cellulose.	Combination glyptal/nitro-cellulose lacquer.	Glyptal-urea synthetic.	Oil modified glyptal synthetic.	Oil modified glyptal synthetic.
Stoving Conditions	—	—	30 mins., 250° F.	30 mins., 250° F.	30 mins., 250° F.
Compositional %					
Volatile Spirit (loss in wt., 3 hours @ 110° C.)	65.3	80.5	60.5	88.3	45.6
Total Solids	34.7	9.5	39.5	41.7	54.4
	100.0	100.0	100.0	100.0	100.0
Physical					
Specific Gravity @ 25° C.	1.013	0.884	1.044	1.039	0.912
Viscosity at 25° C. centipoises	750	15	540	330	540

specimens with phosphate passivation No. 2 retain a sufficient standard of quality to be regarded as satisfactory.

Table III deals with an enamel finish comprising a synthetic coat of stoved synthetic enamel. Without chemical pretreatment the finish is initially poor, but good with chromate or phosphate pre-treatment. Under the durability tests, however, deterioration occurs, so that, again, Samples No. 4, having phosphate passivation No. 2, are the only ones that retain a satisfactory degree of adhesion.

Table No. IV refers to panels using the same enamel finish, but in conjunction with a stoved synthetic zinc chromate primer. With this, again, Samples No. 4, with the phosphate passivation No. 2, are the only ones that

withstand the test periods satisfactorily, all the others being very poor.

Table No. V covers a similar series of panels, only the primer is a red oxide synthetic, with the synthetic enamel finishing coat. Initially, only the samples having chromate passivation, or phosphate passivation No. 2, are really good. Under the humidity test, the chromate passivated samples deteriorate appreciably, but under the atmospheric test, both those with chromate passivation and phosphate passivation No. 2 remain sound. Here again, therefore, the phosphate passivation No. 2 is outstanding.

The samples from these tests that were still very satisfactory at the end of the 30-day test period, were left under the test for a further period to a total of 6

months and then re-assessed. They all comprised samples having phosphate passivation No. 2.

Results are briefly given in Table No. VI. It is to be noted that in this longer time, the cellulose finish deteriorated and became very poor, but with all the stoved finishes, very little signs of deterioration, if any, were evident. The conclusion drawn from the whole test series is that the phosphate treatment is the only one that really affords permanent benefit in ensuring adhesion.

It may be, of course, that the nature of the cadmium plating and the conditions under which it is deposited have some bearing and it may be possible that work could be done to modify the chromate treatment to give improved results. It is also evident that the type of phosphate treatment cannot be chosen indiscriminately.

The enamel media employed in these tests were normal commercial varieties as shown by the characteristics given in Table No. VII. The cadmium plating was an ordinary dull cadmium plate deposited from a cyanide electrolyte at about 10 amps./sq. ft. The thickness of the cadmium was 0.0005 in. min. average, and 0.0003 in. min. local thickness.

The chromate treatment consisted in taking the plated work direct from the washes after plating and treating it by immersion for 10-15 secs. at room temperature in a solution containing 200 grams. of sodium dichromate

crystals and 6.6 mls. of concentrated sulphuric acid per litre of water. The treated work was washed in cold water and air dried at a temperature not exceeding 140° F.

Phosphate treatment No. 1 was applied using a solution of zinc nitrate, sodium phosphate, phosphoric acid and nitric acid, adjusted to a suitable concentration and acidity.

Phosphate treatment No. 2 used one commercial phosphate process having a nitric acid accelerator with immersion time of 3 mins. at a temperature of 180-185° C.

After treatment in these phosphate solutions, the work was washed in hot water and then given a rinse of 15-30 secs. in a solution of chromic acid at 170° F., this chromate rinse being prepared from  $\frac{1}{2}$  lb. of chromic acid in 100 gals. of water.

The proprietary phosphate treatment is an exceedingly simple one to operate and to control and there is no reason to anticipate other than satisfactory results upon all types of cadmium coating. The phosphate film formed, however, is an electrically insulating film, but this is objectionable only in specific instances and, even then, electrical contact can be made by breaking through the film. This type of treatment certainly seems to be more useful than chromate treatments where enamel adhesion is the important factor.

### A New Range of Aerofoil Fans

ONE of the post-war developments in fans has been the introduction of the Series I aerofoil fans by Woods, of Colchester, Ltd., an associate company of the G.E.C. These are of the medium pressure, single-stage, axial flow, non-guide-vane type and are designed for high outputs against resistances up to 1 $\frac{1}{2}$  in. static water gauge. They are available in nine different sizes from 6-48 in. diameter. The range is intended primarily for applications which do not warrant the heavier capital cost of high pressure axial flow fans of the guide-vane type. Their total efficiency is of the order of 65%, which may be regarded as amply satisfactory for the duties covered, since the power inputs are relatively low.

An outstanding feature of these fans is their compactness, as the design permits of comparatively small hub and motor diameters and eliminates the need for upstream and downstream fairings. The impellers, which are cast in one piece from aluminium, have aerofoil section blades of the constant-chord constant-pitch type, and are housed in accurately dimensioned cylindrical casings. They have considerable mechanical strength and are accurately balanced to ensure vibrationless running at all listed speeds. The number of blades varies from four on the 6-in. fan to twelve on the 48-in. size, but the same degree of overall solidity (ratio of blade and hub surface to impeller area) is maintained throughout the range, making each size one of a family.

Three different blade angles are offered in each size which, when used with the range of motor speeds available, provide a choice of fans to suit most duties. A non-overloading characteristic is also incorporated, the maximum power input occurring at approximately the point of maximum static efficiency.

The driving motors, which are manufactured entirely in the factories of Woods of Colchester, are specifically designed for their duties. They are totally enclosed and liberally rated, and have ample power to cope with the

highest pressure which the fan is capable of developing. In order to minimise the possibility of over-heating, the motors have been designed for a temperature rise in the region of only 65° F., as against the rise of 90° F., permitted by the B.S.S. for totally enclosed motors. Buxton tested flameproof motors for operating in inflammable atmospheres can also be supplied on fans from 12-38 in. diameter.

Fan casings of the "long" type entirely enclose the motor, and are fabricated in heavy gauge mild steel which is reinforced at the points of motor suspension and fitted at both ends with angle-iron flanges, drilled for attachment to ducting or other apparatus. Inspection doors, external terminal boxes and external lubricators are provided on all except the two smallest sizes.

The Series I aerofoil fans are becoming widely used for ventilation purposes and industrial apparatus, as well as for agricultural and marine applications. Further information concerning these fans is contained in a recent publication which is available from Woods, of Colchester, Ltd., Braiswick Works, Colchester.

### The Iron and Steel Engineers' Group

THE seventh meeting of the above Group will be held at the offices of the Institute, 4, Grosvenor Gardens, London, S.W.1, in the morning and afternoon of June 22nd, 1948. A buffet luncheon will be held in connection with the meeting. The following papers will be presented for discussion:-

"Electric Control Gear for Steel Mill Auxiliaries," by R. A. West.

"Electronic and Servo-Electronic Controls and their Application to the Iron and Steel Industry," by Dr. W. G. Thompson.

"The Maintenance of Electrical Machinery in Iron and Steel Works," by G. S. Martin and M. Y. Harvey.

# Some Modern Developments in Steels for Welded Structures

By W. Barr, A.R.T.C., F.I.M.

Chief Metallurgist, Messrs. Colvilles Ltd., Motherwell

*Demands of engineers for an easily weldable constructional steel of high tensile strength have been persistent for many years and research has been continuous in efforts to meet this need. Certain characteristics of steel have assumed importance where welding is concerned, and these were discussed by Mr. Barr in a recent lecture before the Belfast Association of Engineers. Many aspects of this subject will be of considerable interest to readers and we are indebted to Mr. Barr for permission to present his lecture.*

WHEN structural work was carried out by the smith, welding was largely confined to wrought iron and soft mild steels. For the high carbon steels the smith needed to be an expert to avoid burning the metal when heating it to the higher temperature necessary for welding. With the introduction of the metallic arc and resistance processes, welding became no longer restricted to this narrow range of steels. Fresh problems were encountered with these developments; certain characteristics of steel assumed importance, one in particular being the hardening of the parent plate adjacent to the weld deposit, and it is proposed to consider these characteristics in the light of recent experience with a view to the solution of problems encountered.

## Weld Hardening

The heat generated in the arc momentarily raises the parent metal at the weld boundary to a very high

temperature. On subsequent cooling, the comparatively large mass of surrounding cold metal conducts heat away rapidly, thus acting as a powerful quench. The effect is shown clearly in Fig. 1, representing sections cut through single run deposits laid on high tensile plates. After polishing and etching, the sections have been subjected to a Vickers diamond hardness Survey the hardness values being inserted on the micrographs. It will be observed that the maximum hardness values are obtained on the parent plate in a narrow zone immediately adjacent to the weld deposit.

For a given steel, the maximum weld hardness is governed by the speed of cooling, which depends upon the heat input in relation to the thickness of the section being welded. Apart from pre-heating, the heat input can be governed, within limits, by the gauge of the electrodes used. The relationship between electrode gauge and plate thickness for a steel containing 0.25%

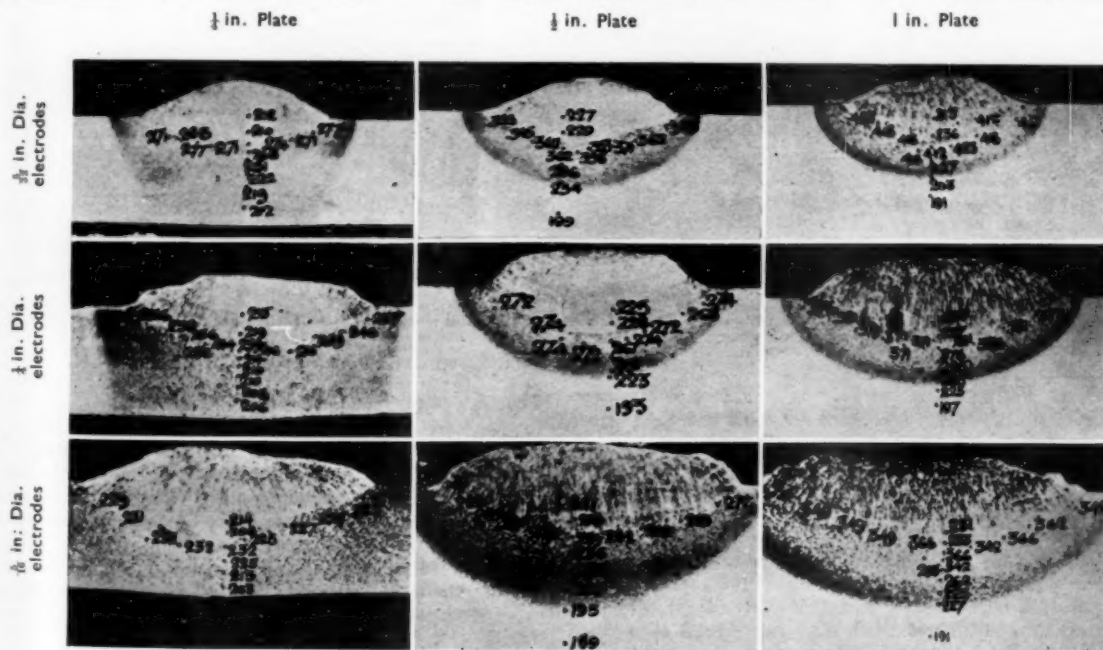
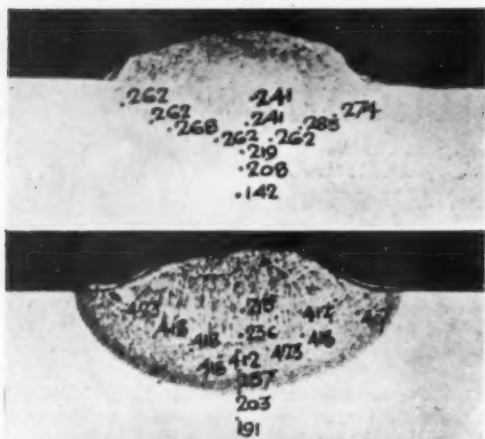


Fig. 1.—Influence on weld hardening of electrode size and plate thickness.  
(High tensile steel 0.25% C. 1.60% Mn.)



Mild steel (0.21% C, 0.70% Mn) 1 in. Plate— $\frac{1}{8}$  in. dia. electrode



High tensile steel (0.25% C, 1.60% Mn) 1 in. Plate— $\frac{1}{8}$  in. dia. electrode.

Fig. 2.—Influence of composition on weld hardening.

carbon, 1.6% manganese, is shown in Fig. 1, and in the table below, which clearly demonstrate the marked decreases in weld hardness in this type of steel when using large gauge electrodes.

Electrode	Plate Thickness	Mean V.D.H.	Boundary Zone
$\frac{1}{8}$ in. dia.	$\frac{1}{8}$ in.	272	
$\frac{1}{8}$ in. dia.	$\frac{1}{8}$ in.	256	
$\frac{1}{8}$ in. dia.	$\frac{1}{8}$ in.	228	
$\frac{1}{8}$ in. dia.	$\frac{1}{8}$ in.	357	
$\frac{1}{8}$ in. dia.	$\frac{1}{8}$ in.	277	
$\frac{1}{8}$ in. dia.	$\frac{1}{8}$ in.	271	
$\frac{1}{8}$ in. dia.	$\frac{1}{8}$ in.	416	
$\frac{1}{8}$ in. dia.	$\frac{1}{8}$ in.	378	
$\frac{1}{8}$ in. dia.	$\frac{1}{8}$ in.	355	

The effect of composition on weld hardening is illustrated in Fig. 2, representing sections through a single run deposit using a  $\frac{1}{8}$  in. dia. electrode on a 1 in. plate of mild steel and high tensile steel respectively.

### Weld Cracking Tests

Excessive hardening of the parent plate will obviously be associated with lack of ductility in the welded joint, and is likely to lead to cracking in restrained joints. For this reason, various forms of cracking tests have been devised. Perhaps the best known is that introduced by Swinden and Reeve,\* see Fig. 3. In this test the plates, usually  $\frac{1}{2}$  in. thick, are bolted securely to an underlying slab to prevent buckling. Three edges of the plate are fillet welded and the assembly thereafter allowed to cool to room temperature. The fourth edge is then welded with the chosen electrode and technique under test. The test weld, therefore, cools under conditions of the most severe restraint. Subsequent examination of polished sections cut through the test fillet reveals any cracking which has taken place. Fig. 4 illustrates the Reeve test results obtained on a steel containing 0.25% carbon and 1.6% manganese, using small and large gauge rods respectively. Note the crack in the

vertical leg of the fillet with the small gauge rod and the absence of cracks with the large gauge rod.

Where multi-run joints are subjected to little or no restraint, cracking is not likely to occur. In such joints weld hardening is much reduced, due to the annealing or softening action of the superimposed runs on the preceding runs, and to the joint heating up as welding proceeds, which produces an effect similar to pre-heating for a single run joint.

### Low Alloy High Tensile Structural Steels

Excessive weld hardening does not arise in straight carbon steels until the carbon content exceeds about 0.30%, hence the absence of trouble from this cause in

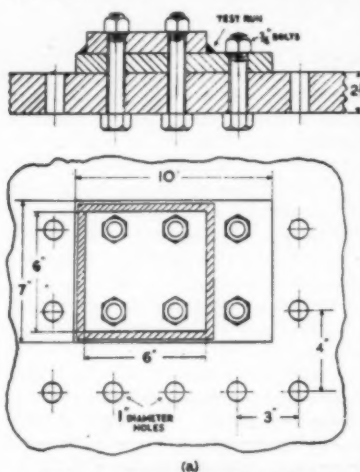
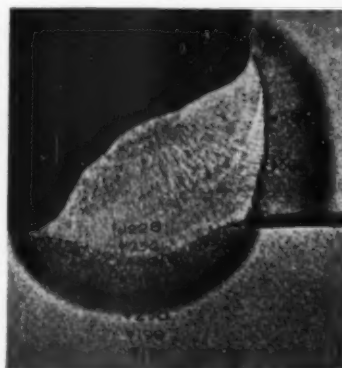
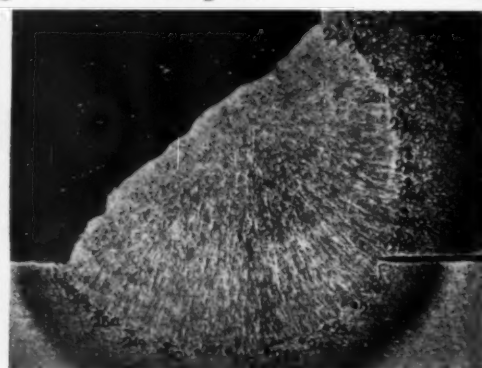


Fig. 3.—Reeve cracking test.



$\frac{1}{8}$  in. dia. electrode.



$\frac{1}{2}$  in. dia. electrode.

Fig. 4.—Effect of electrode gauge on cracking of high tensile steel.

direction by heat treatment, recourse has had to be made to alloy additions, but here again there is a limit which is governed by the price the engineer can afford to pay for the steel. Manganese, being relatively cheap, is usually selected as the basis for the so-called low alloy weldable structural steels and along with this element there may be additions of one or other of the elements, silicon, nickel, chromium or molybdenum. Taking the simplest steels first, British Standard 968 is specified as follows:—

Carbon .. ..	0.25% MAX.	} Ni + Cr + Mo = 2% max.
Manganese ..	1.8% MAX.	
Nickel .. ..	0.5% MAX.	
Chromium ..	1.0% MAX.	

\* Journal of the Institute of Welding, January, 1938, Vol. 1.



Maximum Stress—35/41 tons/sq. in.

Yield Point—21 tons/sq. in. up to 1 in. thick for plates,  $\frac{3}{4}$  in. thick for bars and sections.

Other steels in the same category are Admiralty "D.W." 35/41 tons tensile, with a limit of proportionality of 17 tons/sq. in. minimum for plates up to 1 in. thick and a carbon content not exceeding 0.23%, a typical composition being 0.21% carbon, 1.4% manganese; also the Bailey bridge steel which was used in such large quantities in the recent war. The specification for this steel is:—

Carbon	Manganese	Yield Point	Max. Stress
0.26% max.	1.3/1.7%	Tons/sq. in. 23	Tons/sq. in. 35/43

A feature of these low alloy high tensile steels is the relatively high yield/tensile ratio, and an example of what can now be obtained in this respect is illustrated by the undernoted list of steels which have been developed by the firm with which the author is associated.

Brand	C. %	Mn. %	Ni. %	Cr. %	Mo. %	Max. Stress Tons/ sq. in.	Yield Point Tons/ sq. in.	Equivalent R.S. Spec.
"Ducol"	0.30	1.7	—	—	—	37/43	23 up to 1 $\frac{1}{4}$ in.	B.S.548
"Ducol"	W.19	0.21	1.5	—	—	34/38	19 up to 1 in.	—
"Ducol"	W.21	0.22	1.7	—	—	35/41	21 up to 1 in.	B.S.968
"Ducol"	W.24	0.20	1.5	0.5	0.25	35/41	24 up to 1 in.	—
"Ducol"	W.27	0.18	1.4	0.5	0.25	35/41	27 up to 1 in.	—

The latest steels of this type, such as Ducal "W.24" and "W.27," have been developed to meet existing demands for high duty service, and a wide field for their application is anticipated when engineers become familiar with their characteristics, and the benefits to be derived from their use. Typical test results for Ducal "W.27" are shown below:—

#### Mechanical Properties of Ducal "W.27"

Plate	Yield Point Tons/sq. in.	Max. Stress Tons/sq. in.	Elongation % on 8 in.	Red. of Area %	Izod Impact Ft. lbs.
$\frac{1}{4}$ in. Length	28.6	39.3	16	47.0	—
Cross	28.5	38.6	15	46.0	—
1 in. Length	28.2	38.1	18	35.0	34, 41, 38
Cross	30.0	36.5	20	42.0	23, 24, 27
$\frac{1}{2}$ in. Length	31.7	40.0	19	48.0	42, 38, 48
Cross	30.0	41.0	19	42.0	25, 30, 26
3 in. Length	27.7	39.3	19	46.0	34, 40, 28
Cross	26.4	38.4	18	43.0	29, 38, 28

A typical Reeve test section from a 1-in. thick plate of Ducal "W.27" welded with  $\frac{1}{4}$  in. dia. electrode is illustrated in Fig. 5. It will be observed that a certain degree of weld hardening accompanies the use of this gauge of electrode from which it is evident that in dealing with such a steel commonsense precautions should be taken, such as the use of larger gauge rods and the avoidance of welding out of doors in cold weather on thicker sections without a modicum of preheating.

Engineering design of steel structures has been based primarily on the maximum or breaking stress, the actual working stress being of the order of one-quarter of the maximum stress. In many instances, however, it is the yield stress which really matters and not the maximum stress. This is certainly true for members in compression and also in tension where the live load is a relatively small proportion of the total load. The rational use of the yield stress as a basis for design is of paramount importance when dealing with high tensile structural steels of the type under consideration, where a high yield stress/maximum stress ratio is a feature. B.S.15 has been amended to specify a yield point of 15  $\frac{1}{4}$  tons/sq. in., the maximum strength

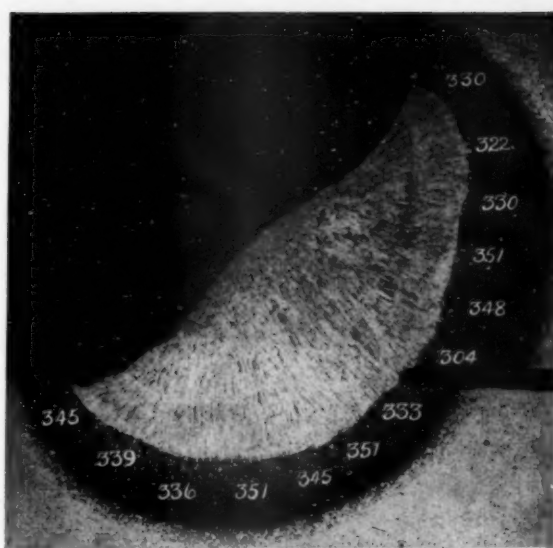


Fig. 5.—Reeve test on Ducal "W.27" steel 1 in. thick plate,  $\frac{1}{4}$  in. dia. electrode.

remaining at 28/33 tons/sq. in. Compared with B.S.15, Ducal "W.27" provides an increase of over 77% in yield stress, whereas on a basis of maximum stress the increase is only 25%. It follows, therefore, that the use of a steel of this type can be profitable only where design is based on yield stress. Only in this way can the saving in weight be made to compensate for the increase in price.

An interesting application of Ducal "W.27" was in the construction of a 120-ft. jib for a 20-ton Morgan derrick crane built by Sir Wm. Arrol and Co. Ltd. The main angles in this jib were 4 in.  $\times$  4 in.  $\times$   $\frac{7}{16}$  in. in Ducal "W.27" steel and the test results obtained on these angles were as follows:—

Maximum Stress Tons/sq. in.	Yield Point Tons/sq. in.	Elongation % on 8 in.
40.5	30.6	17
41.5	30.6	17

It will be observed that the minimum yield point in the above tests was 30.6 tons/sq. in. It was, therefore, decided, as an experiment, to design on a basis of 30-ton yield stress. The bracing consisted of mild steel tubes 1  $\frac{1}{2}$  in. bore with ends flattened and welded to the 4 in.  $\times$  4 in. angles. The jib was entirely welded and the weight of the completed job was 4  $\frac{1}{2}$  tons compared with 9 tons for the normal mild steel construction. This crane, a photograph of which is shown in Fig. 6, has been in service since January, 1947, and no troubles of any kind have been experienced.

Low alloy high tensile steels for welded construction are being used on a large scale in the United States of America, a notable application being in railway freight cars where considerable saving in weight has been effected. One of the most interesting of these American steels, which is now being made in this country, is known as "Cor-Ten," the composition of which, as shown below, is somewhat unusual.

Carbon	0.12% max.
Manganese	0.2/0.5%
Phosphorus	0.07/0.15%
Silicon	0.25/0.75%
Copper	0.25/0.55%
Chromium	0.5/1.25%
Nickel	0.65% max.

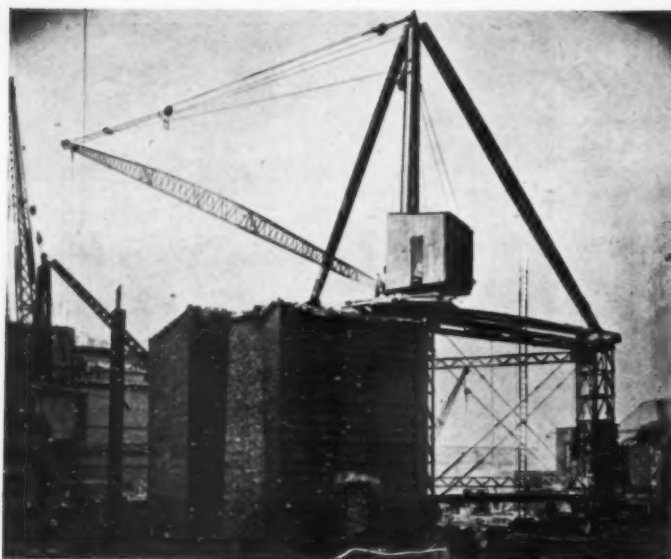


Fig. 6.—20-ton derrick crane with 120-ft. high tensile steel jib.

In plates up to 1 in. thick, this steel gives a minimum tensile strength of 30 tons/sq. in. with a minimum yield point of 21 tons/sq. in. The steel is as easy to weld as mild steel and in addition possesses very good rust resisting properties both in urban and marine atmospheres.

Another important application of "Cor-Ten" is in ship construction. Experience gained to date in America indicates that the principal types of vessels in which the properties of "Cor-Ten" promise the largest saving are tankers handling petroleum products, ships for river, lake and canal service where draught is limited, and freighters in service that permit increasing dead weight tonnage by reducing the weight of the ship structure. The American Bureau of Shipping has indicated that it is prepared to approve "Cor-Ten" for other riveted or welded construction with a general reduction from mild steel scantlings of about 10s. "Cor-Ten" has also been approved by Lloyds Register of Shipping as a special quality steel for shipbuilding.

#### Alloy Steels

Under this heading it is proposed to discuss some of the problems which arise in the welding of armour plate, although the remarks are more or less applicable to high carbon steels and alloy structural steels in general, the latter being usually supplied in the quenched and tempered condition.

In alloy structural steels and armour plate, the purpose of the alloy content—nickel, chromium, molybdenum, etc.—is to increase the hardenability of the steel by decreasing the cooling rate necessary to obtain hardening so that the material will respond to heat treatment in relatively large masses. Unfortunately hardenability is not compatible with weldability. In other words, those characteristics of the steel which are a virtue in heat treatment processes involving quenching are a nuisance in welding because of the excessive hardening which occurs in spite of preheating of the use of the largest gauge of electrode. A manifestation of the same problem is found in gas cutting armour-



Fig. 7.—Butt welded joint in 2 in. thick armour plate using 25/20 austenitic electrodes.

plates when hardening of the edge hinders subsequent machining.

Since weld hardening of such steels is unavoidable, the problem that arises is how to prevent cracking in the hardened zone. Much study has been devoted, in recent years, to the mechanism of this type of crack formation and Hopkins\* has shown that it is due to hydrogen from the electrode deposit diffusing into the hard zone of the parent plate on cooling. There are two methods of combating this: (1) elimination of hydrogen from the deposit by the use of special ferritic electrodes, which have been recently developed for this purpose, and (2) the use of austenitic stainless steel electrodes which, unlike the ferritic material, retains the hydrogen in solution on cooling.

A wide field of application, including the lighter gauges of armour, is anticipated for the special hydrogen-free ferritic electrodes, but where exceptional toughness is required, as in heavy armour which is required to withstand the shock of large calibre attack, there is at present no alternative to the austenitic electrode. Besides eliminating cracking in the hard zone of the plate, the austenitic deposit has the added advantage of much greater notch toughness than can be obtained in a ferritic deposit. An etched section cut through a butt welded joint in heavy armour plate is illustrated in Fig. 7.

#### Brittle Fracture

Under this heading it is proposed to discuss a phenomenon which came into prominence during the late war with the spectacular failure of several American welded ships. These ships, be it noted, constituted a very small part of the great fleet of American ships constructed in United States' yards by the application of electric welding on an unprecedented scale. In such bold wartime enterprise it is not surprising that failures were encountered, some of which attracted much attention due to the catastrophic nature of the fractures, which resulted sometimes in the ship actually breaking in two. A disconcerting feature was the brittle nature of the cracks which spread across the plates with great suddenness in spite of the fact that the same plates had shown perfectly normal ductility in the orthodox

\* Hopkins. Trans. Institute of Welding, July, 1944.

tensile acceptance tests. Tests carried out by the author on specimens cut from some of these fractured plates confirmed that the material had ample ductility as disclosed by tensile and cold bend tests. In most cases the occurrence of these ship failures coincided with a drop in atmospheric temperature to freezing point or below, and there was a tendency in some quarters to regard the phenomenon as something of a mystery. Actually, however, manifestations of the same type of brittle fracture on a much smaller scale are sometimes encountered, especially in cold weather, in engineering shops engaged in the fabrication of mild steel, particularly where such operations as cold flanging or bending are performed. The reason for the greater incidence of such fractures in welded, as compared with riveted, construction appears to lie in the greater rigidity of the welded structure, also in the much greater facility for crack propagation which it affords.

Such fractures are brought about by the presence of stress raisers, such as notches, which are absent in the carefully prepared tensile or bend test used for acceptance purposes. Hence the apparent anomaly that a plate, which has given good values for ductility as measured by percentage elongation or reduction of area, breaks with a brittle fracture during fabrication or in service. Proneness to brittle fracture, which is a characteristic of mild steel as distinct from wrought iron, is referred to as "Notch Sensitivity" and can be assessed only by the use of a notched bar test of which there are various forms. In this country the Izod is the standard form of notched bar test, but, unfortunately, there has been and there still is, controversy regarding the practical significance of the results obtained when the test is applied to ordinary mild structural steel. An important feature of this test is its dependence on temperature. This is illustrated in Fig. 8, which shows the temperature/Izod curves for two specimens of mild steel, one manufactured in the orthodox way as for ship plates (Curve 'A'), and the other made of "fine grained" steel (Curve B). It will be observed that in both cases there is a transition from a tough to a brittle condition occurring over a temperature range the extent of which depends on a number of factors.

Considering first, steel in the orthodox condition (Curve A), the transition temperature, tough to brittle fracture, occurs normally in the vicinity of 0°C. Hence

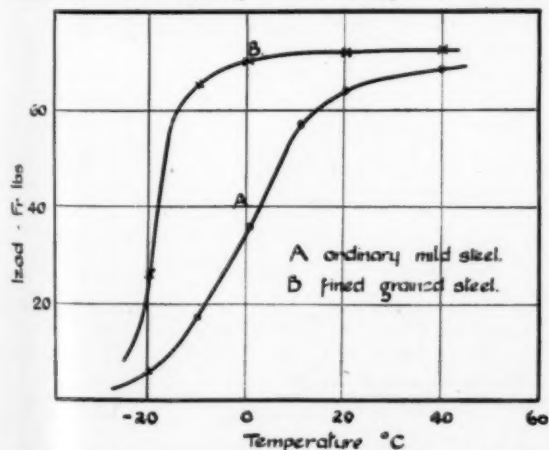


Fig. 8.—Temperature impact curves of mild and fine-grained steel.

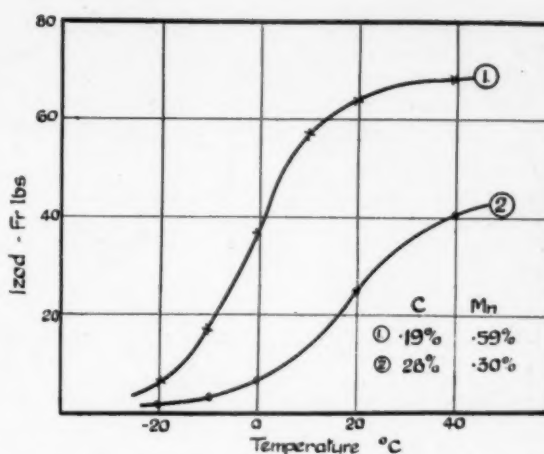


Fig. 9.—Effect of manganese/carbon ratio on temperature impact curve.

the reason for the greater susceptibility to brittle fracture in cold weather. Apart, however, from energy absorbed this transition is apparent in the appearance of the fracture test. In the tough condition, energy is absorbed in the course of the propagation of the crack which proceeds more in the nature of a tear accompanied by considerable deformation, the crystals being drawn out to fine threads which impart a fibrous appearance to the fracture. At the other extreme, fracture takes place by cleavage through the crystals with little or no deformation and so the fracture presents a shiny crystalline appearance. Because of the characteristic difference in the appearance of the two types of fracture there has been a tendency to accept this as the criterion of the test and to discount the energy absorbed.

On this basis the aim should be to provide a steel with a transition range below the temperature anticipated in service, such as is shown for the fine grained steel in Curve B, Fig. 8. This would certainly be the ideal solution, for it would mean that a crack once formed would not propagate in a catastrophic manner, but, unfortunately, in the light of our present knowledge, the use of such a steel would add substantially to the price of ship plates.

The author prefers to deal with the matter less drastically by legislating only for conditions which give rise to the initiation of cracks. Consider, for example, two mild steel ship plates of the same thickness, both of which in the Izod test exhibit similar fractures which are predominantly of the cleavage type, but in one case the energy absorbed in the test is only 5 ft. lb., while in the other case it is 25 ft. lb. Now, since a cleavage fracture absorbs negligible energy in its propagation, it follows that the difference in these two plates lies in the different amount of energy they are capable of absorbing by plastic deformation at the bottom of the notch before the formation of a crack. In other words, the plate with a higher Izod value is less sensitive to the existence of such notches as are difficult to eliminate in the course of ordinary workmanship. Further, it should be noted that a very small amount of deformation will suffice for relief of local stresses besides decreasing the potency of the stress raiser by increasing the radius at the bottom of the notch.



Of the several factors which affect the notch sensitivity of mild steel, perhaps the most notorious is nitrogen, which when present in excess of 0.008% approx. has a notch embrittling effect which is particularly noticeable on ageing after plastic deformation—a phenomenon known as strain-age embrittlement. The particular proneness to brittle fracture of steel made by the Bessemer process is due entirely to its relatively high nitrogen content ( $> 0.01\%$  compared with  $< 0.005\%$  for open hearth steels). In referring to Bessemer steel, it is interesting to note that steps have been taken in recent years to reduce the nitrogen content by a modification of the process. When this has been accomplished in a satisfactory manner there appears to be no reason why Bessemer steel should not hold its own with steel made in the open hearth.

Reverting to the failures in the American welded ships, an exhaustive report of investigations carried out in the United States has been published.<sup>1</sup> The conclusions reached are that faulty design and welding technique were, in the main, responsible for the troubles, but at the same time stress is laid on the need for decreasing the notch sensitivity of ship plates.

With the rapid expansion of welding in British shipyards the problem cannot be ignored, and it is encouraging to note that the subject is being actively pursued by a Sub-Committee of the Admiralty Ship Welding Committee along with the British Iron and Steel Research Association. The author has collaborated in several of these researches, in one of which the beneficial effect of manganese has been demonstrated.<sup>2</sup> Owing to the shortage of manganese in the United States during the war, many of the ship plates manufactured at that time had a lower manganese content than

usual and much of this material found its way into the ships in question. In British practice it is unusual to find ship plates containing less than 0.45% manganese, whereas in these American plates the manganese content was frequently as low as 0.35% and in some cases even lower.

The beneficial effect of manganese on the notch sensitivity of mild steel is shown by the Izod temperature curves for two 1 in. thick plates of approximately the same tensile (30 tons/sq. in.), both plates being in the normalised condition to eliminate such variables as finishing temperature from the rolls, etc. (see Fig. 9).

A further advantage of manganese is its effect in retaining the notch toughness of mild steel which has been slowly cooled from the normalising or rolling temperature, as indicated by the following Izod values on the same two 1 in. thick plates after normalising and annealing respectively.

#### Izod Ft. Lb. at 18° C.

	Normalised	Annealed	
(1) 0.19% C.	62	45 (27½% Decrease)	
(2) 0.28% C.	25	15 (40% Decrease)	
			0.59% Mn. 0.30% Mn.

It should be noted that the test results quoted above are intended to indicate trends and that they represent mean results obtained on plates in the normalised state, whereas ship plates are supplied in the "as rolled" condition. Arising out of these investigations the author considers that the manganese content of steel used for ship plates should not be allowed to fall to too low a level. Since such plates are supplied to a tensile range, the manganese content is best expressed as a manganese/carbon ratio and a practical recommendation is that this ratio should be not less than 3. As an additional safeguard for the thicker plates, say,  $\frac{3}{4}$  in. and over, a normalising treatment may justify the additional cost involved. This, however, is one of several factors which are being investigated at the moment.

<sup>1</sup> "The Design and Method of Construction of Welded Steel Merchant Vessels," U.S.A. Ship Structure Committee, July, 1946.  
<sup>2</sup> "Effect of Manganese/Carbon Ratio on the Brittle Fracture of Mild Steel." (Barr and Honeyman, Jour. I. and S. Inst., Oct., 1947.)

## Plant Engineering Division

Centralised Engineering Research is a comparatively new development in the steel industry, but B.I.S.R.A. has now launched an expanding programme of research through the Plant Engineering Division. This work is divided into the usual classes of engineering mechanical, engineering, electrical engineering, civil engineering and the division is also responsible for all research into fuel technology.

Applied research is the main object and the investigations which have been commenced in the last 12 months are directed towards the improvement of engineering practice in the industry through the application of original techniques or the adaptation of known techniques to the special requirements of iron and steel making. This implies the improvement of operational methods as well as the use of new standards of plant design and performance and both those requirements are considered in the programme of research.

The large scale reconstruction schemes which the industry is now commencing provide an unusually wide scope for the application of new methods and techniques and the majority of engineering problems at present being investigated indicate a close connection with this reconstruction programme. These projects include:—

Design of melting shops. Layout of works, buildings, plant and services. Improvement of electric overhead travelling cranes. Economics of locomotives, rolling stock and traffic improvement of open-hearth furnace charging methods. Melting and liquid fuel flow.

Other investigations have long range objectives, these projects include:—

Survey of mill furnaces. Survey of fuel distribution and usage in unit processes. Application of gas turbines to blast-furnace blowing. Utilisation of sensible heat in coke (e.g., dry cooling). Application of servo mechanisms and remote indications. The engineering aspects of improved blast-furnace burden distribution

## Locomotive Works Practice

THE Locomotive Manufacturers' Association of Great Britain announces that to enable detail design and manufacture of all types of locomotives and spare parts to be carried out at individual factories in an identical manner, and thus provide standardisation and greater interchangeability in service, the whole locomotive industry in Great Britain has adopted a comprehensive standard works practice for limits and fits. Full details will be included in a book to be issued by the Association.



# Melting Furnaces in German Steel Foundries

By Dr. Ing. Hans Stein and Dr. Ing. Karl Roesch, Remscheid

*This is a report on trends in Germany of the various types of steel-making furnaces used in steel foundries. It was prepared by the authors at the request of the Steel Castings Division of The British Iron and Steel Research Association and made to the Economic Inspectorate of the Control Commission, to whom the Association is indebted not only for arranging its preparation but also for its translation. Readers will appreciate that present conditions in the German industry are somewhat fluid.*

THE following types of furnace are used in German steel foundries for the melting of steel for castings:—

1. Open-hearth furnaces, acid and basic.
2. Bessemer converters.
3. Electric-arc furnaces, acid and basic.
4. High-frequency furnaces, acid and a few basic.
5. Graphite-bar furnaces, acid and neutral.
6. Rotary furnaces, fired with coal dust and oil.

The majority of the furnace systems in steel foundries are governed on the one hand by the diverse requirements in the quality of steel castings put forward by consumers, and on the other by the raw material position in Germany. The percentage proportion of the various steel casting production processes has also been decisively influenced by developments in power, pig iron and scrap prices which to some extent pursued a different course from those in other countries.

About twenty years ago, three or four types of steel for castings proved sufficient, and these were hardly ever alloyed. But with the development of light construction and the necessary lowering of the safety factor which this entailed, the requirements put forward for uniform quality of the cast-steel parts rose higher and higher. It was the power engine, locomotive, wagon building and motor truck industries, in particular, which gave impetus to this rise. To meet these higher requirements, cast-steel parts with their diminished wall thickness had to be entirely crack-free and to possess satisfactory uniform tensile properties at every point. To fulfil this high standard of requirements, steel for castings had to be melted in those furnaces which guaranteed production of crack-free material of uniform composition.

The important and interesting experiments of Professor Thum on resistance to vibratory stresses showed that a steel casting is safer than a welded construction, particularly if attention is given to adequate resistance in transitional cross-sections to vibratory stresses. Then

came the demands of the chemical industry and furnace makers for materials resistant to acid and hammer scale, the melting of which called for special melting units as did the numerous other special alloys.

The development of light metals also compelled the steel founder to produce steels with higher tensile strength and ductility and this led to the discovery that heat-treated alloyed steel castings are lighter in weight than light metal castings of equal constructional strength. As a result of the foregoing developments, the steel foundries in the British Zone set up the furnace units given in Table I which enabled them to produce satisfactory castings to consumers' requirements. To these furnaces must be added an open-hearth capacity of about 175 tons for a number of foundries which draw their steel from associated open-hearth steelworks.

The first steel castings in Germany were made by Bochumer Verein about the year 1850, who were followed soon after by Krupp. The steel for these castings was melted in crucibles.

After the crucible furnace came the open-hearth furnace and later the Bessemer converter for the production of steel castings. The first electric furnace in Germany was started in 1906 at the Richard Lindenberg Steelworks in Remscheid. This electric arc furnace, one of the first in the world, is now in the local museum at Remscheid.

Figs. 1 and 2 show the growth of steel-melting furnaces for steel castings in the British Zone from 1925 onwards in terms of ton capacity. The curves show that while the production of acid open-hearth and Bessemer steel for castings has remained practically constant, production of basic open-hearth cast steel has fallen and that of electric steel has risen considerably. From about 1930 onwards, with the development of acid and heat-resisting steel castings, the high-frequency furnace has also taken part, even though on modest lines, in the total expansion of steel castings. Compared with

TABLE I.—FURNACES ONLY FOR STEEL CASTINGS IN THE BRITISH ZONE OF GERMANY

Rated sizes in tons . . .	0.2	0.3	0.5	1	2	3	4	5	6	7	8	9	10	15	20	25	30	35	40	Total Furnaces	Total Capacity
O.H. . . . .							1	1		2	4		1	10	4	8		2	1	34	605
Electric arc furnace . .						18	2			1	4		4	1						53	280
H. Fr. furnace . . . .						7			16											24	35
Graphite resistance furnace . . . . .	6	3		3																12	6
Bessemer . . . . .				1-5t																84	187
				25	56	3															

With  
80 cupolas

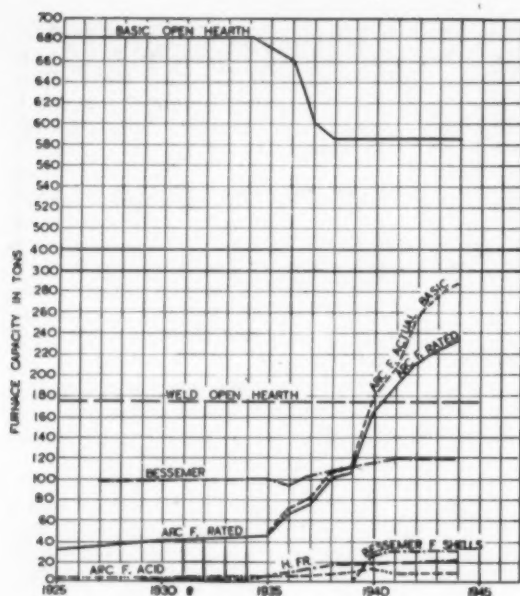


Fig. 1.—Furnace capacity in tons. British zone.

England and America the proportion of acid lined open-hearth and electric-furnace units in Germany is remarkably small.

Fig. 3 indicates the total number of electric furnaces set up in the British Zone.

TABLE II.—PRODUCTION BREAKDOWN OF LIQUID STEEL IN THE DIFFERENT GERMAN ZONES

Zone	Year	O-H	Elec.	Bess.	Total	Year	O-H	Elec.	Bess.	Total
British .. .. .	1942	37	26	37	55	1943	32	28	40	49
American .. .. .	"	28	19	53	9	"	28	19	53	11
French .. .. .	"	8	39	53	7	"	7	34	59	9
Russian .. .. .	"	57	28	15	29	"	53	32	15	31
Total .. .. .	1942	40	27	33	—	1943	36	29	35	—

The growth in the total production of steel castings expressed in 1,000 tons in the former Reich territory is shown in Fig. 4.

The percentage development in steel castings produced in the various furnace systems from 1925 onwards in the former Reich is shown in Fig. 5.

Table II gives a percentage breakdown of liquid cast steel production for the British, American, French and Russian Zones for the year 1942-43.

### 1. Open-hearth Furnaces

As Table I shows, the capacity of most open-hearth furnaces in independent steel foundries ranges between 15 and 25 tons. Of these furnaces 90% are basic-lined. Acid-lined open-hearth furnaces only produce castings for low strengths which can be cast as a rule in green sand moulds. The manufacture of standard steel castings from open-hearth steel was given up in Germany many years ago, as due to the inferior scrap conditions in Germany this steel is too susceptible to heat cracks and flaws.

The percentage production of acid and basic open-hearth steel in terms of total crude steel production in Germany for the years 1935, 1936 and 1937 was as follows:

		Open-hearth Steel Percentage of Total Crude Steel Production	
		Acid	Basic
1935 .. .. .	..	10.5	55.6
1936 .. .. .	..	9.6	51.4
1937 .. .. .	..	9.8	47.6

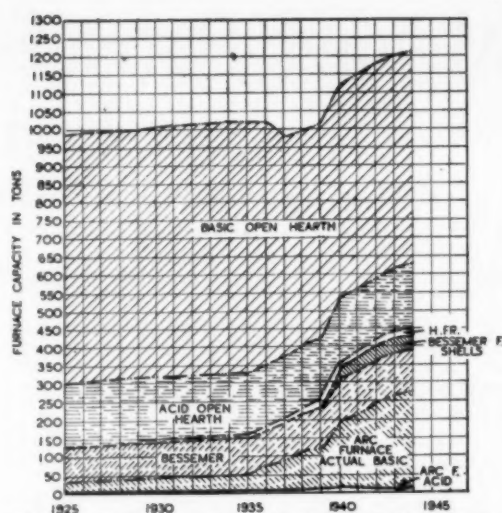


Fig. 2.—Furnace capacity in tons. British zone.

In England the conditions were almost completely reversed.

The basic open-hearth furnace has maintained its lead in the production of medium and heavy steel castings. Control even if limited, of the phosphorus and sulphur contents renders this steel less susceptible to heat cracks if good scrap is charged.

Nevertheless, the percentage proportion of open-hearth steel production has fallen in Germany and other countries for a number of reasons.

For numerous thin-walled and bulky castings this steel is unsuitable as it cannot safely be melted to a sufficiently high temperature to render it non-susceptible to heat cracks. Above all, high melting temperatures cause difficulties when it is necessary to fill hand ladles with cast steel from the furnace ladle. If charges of different composition have to be poured daily, then even in times of normal employment the order programme for large heats causes difficulties with small castings, particularly in continuous production. Both from the economic

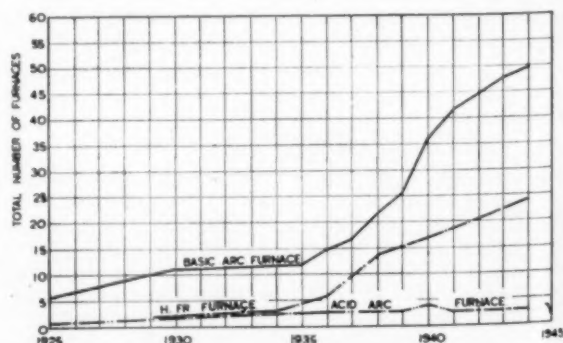


Fig. 3.—Total number of electric furnaces in the steel foundries of the British zone.

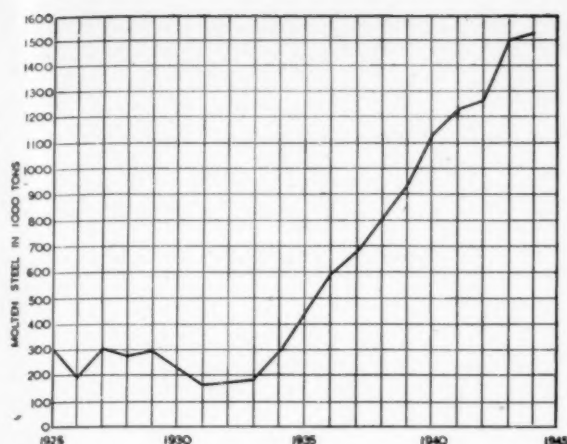


Fig. 4.—Annual production of molten steel for steel castings in Germany.

and heat-economy point of view, the fact must be taken into account that the open-hearth furnace only works satisfactorily above a certain cubic content. Keeping the furnace warm at week-ends entails additional costs as few steel foundries manufacture ingots.

In the extension of existing steel foundries, the large amount of space required by an open-hearth furnace was also a handicap on occasions.

For certain alloyed steel the open-hearth furnace is ruled out on account of the high melting loss in alloying elements.

## 2. Small Converter

Although total production is increasing, the proportion of Bessemer steel castings has not risen. The temporary increase in output at the beginning of the war was due to the manufacture of cast steel shells.

It is known that small castings can be manufactured in green sand moulds from this steel more easily and in many cases more cheaply than by other melting processes, largely because of its great fluidity and the high viscosity of the slag. Moreover, the small converter is highly adaptable for variations in production. The fact that, despite these advantages, this method has not been further developed in Germany is probably due to the scrap and coke conditions being very much inferior to those in other countries. For this reason, Bessemer steel is only suitable for the manufacture of certain products. Steel foundries with electric as well as open-hearth furnaces generally use the converter for the manufacture of green sand castings only up to about 100 kg. weight

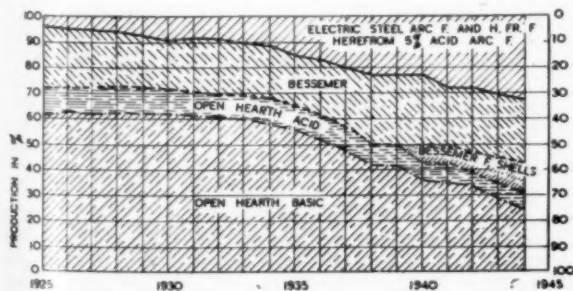


Fig. 5.—Production of steel castings in different types of furnaces in Germany.

per piece. This does not alter the fact that independent Bessemer steel foundries do on occasion manufacture simple castings up to about 1,500 kg. weight.

In the post-war period the Bessemer plants in the British Zone have had serious difficulties to contend with as the following particulars show:

The hematite at the disposal of the Bessemer plants contains about 0.10-0.12% phosphorus. Reactivity and sulphur content of the melting coke are both very high. Even with desulphurisation of the iron in the lander, a content of 0.09-0.13% phosphorus and 0.07-0.09% sulphur must be reckoned with in the finished product. Such steels are highly susceptible to heat cracking so that even the manufacture of standard castings is difficult. Apart from this susceptibility to heat-cracking, the ductility of the steel, particularly in the harder qualities, leaves much to be desired. Recrystallisation in the annealing takes place either incompletely or else very slowly. Annealing temperatures of about 980° C. are, therefore, essential, but this causes heavy scaling on the castings and in the case of mild steels with less than 0.15% C it is frequently impossible to refine the texture by annealing. In addition, the present serious shortage of ferro-silicon renders it almost impossible to blow the steel at a sufficiently high temperature to run off small, thin-walled castings.

Experiments with basic-lined Tropenas converters show that in contrast to the basic Bessemer process the N content of the steel is low. The process, however, has not been adopted to any great extent as the casting properties of the basic steel are perceptibly poorer than that of acid converter steel.

## 3. Electric Furnaces

The power supply system in Germany enables steel foundries to obtain sufficient power at low cost for steel production. The price for melting steel in electric furnaces of 8-12 ton capacity was consequently only about 10-15% higher than the price for liquid steel from open-hearth furnaces of 10-15 ton capacity.

Before the war prices for liquid steel in one well-known German quality steel foundry were about the following level:

10 ton open-hearth furnaces	.. .. .	90.00-100.00 RM/t
8-12 " basic electric furnaces	.. .. .	105.00-110.00 "
3-4 " " "	.. .. .	120.00-140.00 "
3-4 " Acid " "	.. .. .	110.00-120.00 "

The higher price of electric steel compared with open-hearth steel was more than compensated for by its advantages so that, in the end, the electric steel casting was cheaper than the open-hearth casting.

The main reason for the swing-over from open-hearth to electric steel castings was the low susceptibility of basic electric steel to heat cracking. As one has to reckon on a higher content of phosphorus and sulphur in scrap in Germany than in other countries, the casting as required by the engineering industry causes serious difficulties.

Reasons of a technical nature in moulding also contributed largely to the change in the furnace system. Unlike the countries abroad, the sands at the disposal of the German steel foundryman do not permit the casting of large items in sand moulds, which led to the development in Germany of moulding with refractory masses. This is a high-grade refractory moulding material with the disadvantage, however, that it dries very hard. This again means a greater risk of cracking and makes the use of basic electric steel essential for many constructions.



Like open-hearth furnaces, electric-arc furnaces in Germany are practically all basic-lined. Only three small acid-lined electric furnaces were in operation throughout the Reich. In the case of two of these acid-lined furnaces this method was possible only because there was a sufficiency of low phosphorus and sulphur scrap available for them in the form of runners from basic electric furnaces. As this particular works also had at its disposal good small scrap from the machine tool industry, it was successful in being able to charge both acid-lined electric furnaces for the production of small steel castings. In the British Zone out of a total of 50 electric-arc furnaces only three are acid-lined; and of a total furnace capacity of 250 tons only 10 tons—i.e., 4% of capacity is acid. This represents 2% of total capacity for the entire Reich. Developments have thus pursued a different course from those in America.

Further, the increasing use of alloyed steel for the casting of high-duty, oil-quenched and tempered parts has necessitated the use of electric-arc furnaces with which the recovery of the alloying elements, particularly with the double slagging method, is appreciably higher than in the open-hearth furnace.

The introduction of numerous special qualities to meet the increasing requirements of the engineering and motor-vehicle industries also necessitated the casting of numbers of small parts of varying composition. These requirements are best fulfilled by the electric furnace and, as Table I shows, the 3 t and 5-6 t electric furnace were mostly introduced.

In the new standards for electrodes, allowance was accordingly made for small furnace units required by the steel foundries. (See Table III).

TABLE III.—STANDARDISATION OF ELECTRIC ARC FURNACES IN GERMANY IN 1942

FIXED DATA					
Size of Furnace	Dia. of Furnace mm.	Transformer Energy kVa	Electrodes $\phi$ mm.		
			Graphite	Carbon	
L 0.5 .. .. .	1,600	500	150	—	
L 1.6 .. .. .	2,200	800	150	—	
L 3 .. .. .	2,600	1,200	200	—	
L 6 .. .. .	3,400	2,000	250	350 up to 6,600	
L 10 .. .. .	3,800	3,500	300	500	
L 16 .. .. .	4,300	5,000	350	550	
L 25 .. .. .	5,200	9,000	500	—	
L 40 .. .. .	5,700	12,000	500	—	
L 60 .. .. .	6,000	15,000	500	—	

APPROXIMATE DATA					
Size of Furnace	Pitch Line $\phi$ mm.	Voltage Range V	Plate Thickness		Distance from Furnace Casting Level to Roof
			Shell mm.	Bottom mm.	
L 0.5 .. .. .	400	140/80	10	10	800
L 1.6 .. .. .	600	150/87	13	13	900
L 3 .. .. .	700	165/87	15	15	1,000
L 6 .. .. .	950/850	180/90	20	20	1,100
L 10 .. .. .	1,150/1,050	200/90	20	20	1,200
L 16 .. .. .	1,300/1,200	220/90	23	28	1,300
L 25 .. .. .	1,400	140/90	26	28	1,450
L 40 .. .. .	1,500	270/110	28	32	1,550
L 60 .. .. .	1,700	270/110 (300)	28	32	1,600

Tilting angle for all furnaces: Taphole side, 42°; charging door side, 15°.

Basket charging: Size L 6 and upwards only.

Compared with the open-hearth furnace the electric furnace has the advantage of a higher yield per ton furnace content. With the open-hearth furnace one must reckon with a non-operational period of at least three months for repairs, whereas with electric furnaces

the loss of time is very small. Roof changing is quickly effected and only three days are needed to re-line the furnace casing. At one works this period was considerably reduced in the case of a 3 t furnace by resting the bath in two trunnion pivots. This enables the workers to put in a freshly-lined bath in 2½-3 hours including removal and replacement of the electrode holders.

Lastly, the electric furnace can be set up anywhere in the steel foundry which is not the case with the open-hearth furnace.

In recent years the Crespi hearth has been introduced with success in basic electric furnaces. The hearth is made of fine-grained, tar-free dolomite by a special tamping process. Once fired, these hearths prove highly resistant and reduce basic lining costs.

The reasons given above for the growing use of the electro-smelting process have led to similar developments in other countries.

In a comparison of the advantages of acid and basic smelting in an electric-arc furnace, the following conclusions were reached:

(a) *Advantages of acid electric-arc furnace*

1. Lower price of steel.
2. Lower lining costs.
3. Lower consumption of power.
4. High fluidity of steel.
5. High steel yield.
6. High slag viscosity facilitating the pouring of small castings with hand ladle.

(b) *Advantages of basic electric-arc furnace*

1. Low susceptibility of steel to cracking.
2. Low melting loss of alloying elements.
3. High quality of steel even when low-grade scrap is used.
4. High adaptability to consumers' special requirements.
5. Low consumption of electrodes.

#### 4. High-frequency Furnace

With the development of special steel castings for acid and heat-resistant parts, such as magneto castings, etc., the number of high-frequency furnaces has increased rapidly since 1930. Towards the end of the war there were about 20 high-frequency furnaces in operation in steel foundries in the British Zone, but not all of these furnaces possess separate current converters. In contrast to the electric-arc furnace, therefore, they cannot all be charged for the production of steel castings. Most of the furnaces have a capacity of 500 and 1,000 kg. Latterly, high-frequency furnaces were built with a taphole. Two important advantages in this construction are the abolition of the tilting gear and the low consumption of energy since the furnaces are completely detached and consequently there is no loss of electric current. In addition, the robust construction of the furnace permits of it being basic-lined, there being no cross currents to tear the sensitive basic lining.

As no metallurgical slagging takes place in a high-frequency furnace, non-alloyed steel from this type of furnace is inferior in quality to that from the electric-arc furnace.

#### 5. Carbon-bar Furnace

During the final years of the war the graphite-bar furnace was successfully introduced in place of the high-frequency furnace. The furnace consists of a cylindrical drum in the axis of which is a graphite bar. This is

heated with a voltage of about 40 volts up to 2,000–2,500°. The heat radiated against the roof is supplied by a rocking motion to the bath. As the furnace can be sealed easily, current consumption even in small furnace units is relatively low. Graphite-bar consumption amounts to only 3–4 kg. compared with 8–9 kg. per ton in the electric-arc furnace. Due to the highly reducing atmosphere above the bath, the melting loss of alloying elements is very low.

In contrast to the high-frequency furnace, there is more freedom of choice in the lining, there being no fear of a break-out and hence no coil destruction. For special stresses a lining of smelted corundum has proved highly satisfactory.

Initial costs are low. A 1,000 kg. furnace with a reserve drum cost only half of a high-frequency plant of similar capacity.

There is one drawback, however; small pieces only can be charged and only one phase of the circuit can be loaded during operations.

It is quite likely, however, that this type of furnace which is well constructed throughout will replace the high-frequency furnace up to a capacity of 1 ton, as it is very suitable for the manufacture of highly alloyed castings.

The power consumption of the different types of electric furnace can be compared from the curves in Fig. 6. In this connection it must be borne in mind that with the basic electric-arc furnace the method of smelting is of some importance. With acid electric-arc furnaces the low heat conductivity of the lining reduces the consumption of power. The low power consumption of the high-frequency furnace and of the graphite-bar furnace is mainly due to the fact that it is a question of melting the charges only with no slagging. The power consumption of the graphite-bar furnace is remarkably low. An attempt has also been made in Fig. 6 to indicate the range of the most favourable furnace sizes for steel castings.

## 6. Rotary Furnaces

We cannot conclude without some mention of the oil-fired rotary furnace which has found temporary use in a number of steel foundries. The furnace of 1–2 ton capacity is suitable for the manufacture of small steel castings. Oil consumption which is about 40% of the yield of liquid steel can be reduced to 32% by installing good air heaters. The satisfactory fluidity of the steel and the fact that it can be readily cast with hand ladles deserve mention. In this respect this steel resembles converter steel.

Acid and heat-resisting castings can also be made in the rotary furnace.

Production per ton capacity is very good as the furnace vessel can easily be exchanged for another by crane.

## Summary

Development of the various types of furnace is first traced from available statistics. In contrast to America and England, the acid furnace can seldom be used as scrap and pig iron conditions in Germany are unfavourable. In general, however, furnace types and sizes have developed on similar lines to those in other countries.

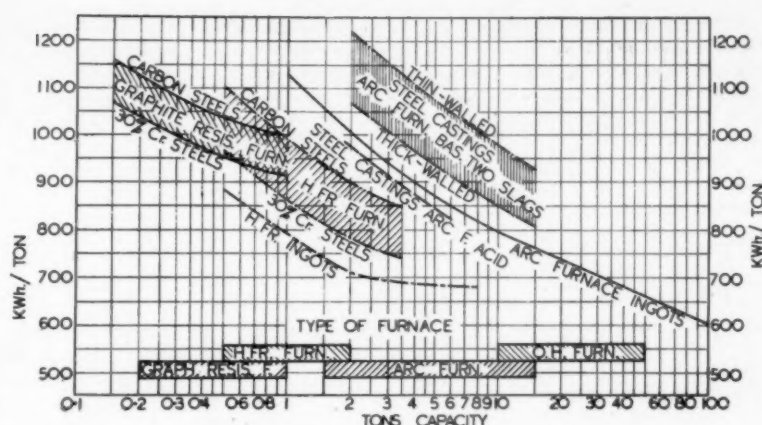


Fig. 6.—Power consumption/ton of steel—medium production.

In the second part, the different types of furnace are described from the point of view of their suitability for the steelfounder. In Germany an innovation is the graphite-bar furnace which may possibly replace the high-frequency furnace up to 1 ton capacity for the production of special highly alloyed steel castings. As part of our statistical records were burned, it has not been possible in every case to give a full statement.

## Radium Technique Developed to Service Industry

FAR-SIGHTED executives in industry and commerce have been quick to realise the possibilities resulting from atomic research. One of the first of advantageous developments arising from this work is the application of gamma rays over a wide field of industry for the non-destructive examination of all metals and metal components and structures with a view to the location and identification of internal flaws which may be present. Although all uranium and radium supplies are controlled by the Government, facilities have been granted to Gamma Rays Ltd. which enable the latest gamma ray applications to be at the disposal of industry.

Gamma rays differs from X-rays in certain fundamental particulars, but, in general, may be regarded as supplementary to rather than competitive with them. Gamma rays, however, are a logical development of radiography and represent a considerable advance with regard to portability and depth of penetration. Clear internal photographs of metal sections up to 9 in. thick can be obtained and the radium sources used by the Gamma Ray service are so constructed that they can be used in positions totally inaccessible by any other means of inspection.

For castings, especially those for key components that require to be 100% sound, this method of inspection will be invaluable to designers and engineers. For the examination of welded structures too, and indeed all metal components in which soundness is an essential requirement, the value of this method of inspection cannot be over-emphasised. With this method there is no need to dismantle a structure to be examined, the equipment using gamma rays is portable and can be taken anywhere for an on-the-spot inspection of the suspected section.

# Exhibition of Zinc Alloy Die Castings

*A recent exhibition of die castings, held in Birmingham, emphasises the progress made in this section of industry. Although it attracted many visitors many would be unable to attend, and this brief report is intended to indicate to those interested in the use of die castings the possibilities they offer.*

**T**HE Zinc Alloy Die Casters' Association have an enviable reputation for the way in which they have stimulated the development of die castings in many industries. The Association was formed during the war to assist the production of munitions by co-operation between British die casters. After the war the function of the Association changed to the furthering of development and technical progress. Queries from all parts of the country are handled anonymously by the Technical Committee, formed of experienced die casters. The Publications Committee is responsible for the preparation of literature, advertisements, lectures and bulletins.

This Association recently held an exhibition of zinc alloy die castings at the Midland Hotel, Birmingham, which was exceedingly well attended. A minor criticism of this exhibition is that a larger hall would have enabled the exhibits to be shown more advantageously, but it is probable that this will be remedied on future occasions.

The pressure die castings were grouped into five sections, with an additional stall containing literature for which the Association has been responsible. One of the most spectacular groups was that dealing with heavy die castings. The large body of a petrol pump meter (Fig. 1) has a multitude of large and small cored holes in six directions. The only machining operations necessary being those of the cylinder bores, crankshaft bearings and valve seatings. The die casting is produced in B.S.1004a, and is heat-treated to ensure the closest dimensional stability.

The gear case of the hotpoint electric washer, and wringer (Fig. 2) attracted much interest. This die casting is 24 in. long and weighs 14 lb. It contains bearing bushes and two motor supporting brackets

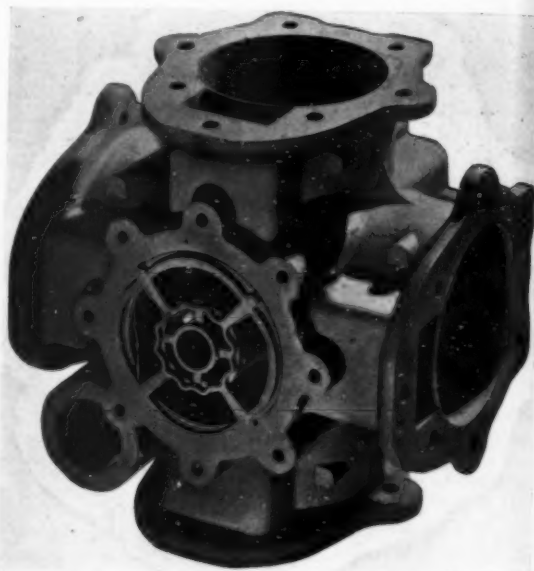


Fig. 1.—Large body of a petrol-pump meter.

included as inserts. Another large casting, illustrating an interesting use of inserts, was a hospital bedside table stand. This had a large die-cast base, from which projected a long steel insert, forming the upright portion. From this, another steel insert formed the horizontal arm, the two being connected by a small zinc alloy pressure casting. This exhibit caused surprise to those who previously had thought of inserts in die castings as being small components only. Yet another example of enterprise was to be seen in two die-cast stators, with laminations included as inserts. On the same stall the upper and lower rings of a side drum were on view.

The exhibits of small die castings included many toys, showing the fidelity of reproduction possible by the use of the process. Some of the castings in this group illustrated the thin sections which can be achieved, as for example, an ammeter case which was only  $\frac{1}{16}$  in. thick.

Fishermen would be interested in an assembly of die castings used in the Elarex fishing reel. The parts fit precisely together; lettering incorporated in the casting shows line capacity and an index of braking degrees. The group illustrating die castings of medium weight contained many examples from the motor trade, including the parts of the well-known Austin radiator grille assembly, showing how a large grille can be made from

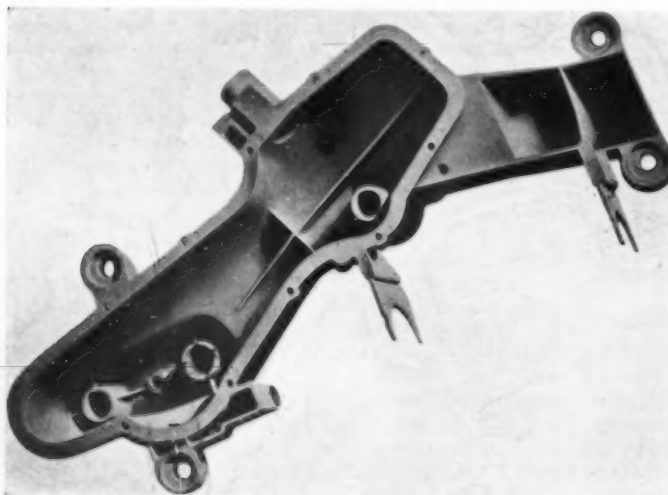


Fig. 2.—Gear case for an electric washer.





Fig. 3.—Hack-saw handles have flawless surfaces.

medium-sized and easily-handled die castings. A fine example of die-casting technique was the Lucas two-toned horn. Also included were parts of cotton-spinning machinery, gears, wringer parts and the crank case of a motor mower. Two die castings in this section, used by a well-known maker of hack-saws, impressed the writer as possessing an almost flawless surface appearance (Fig. 3). Although pressure die castings in this country have for many years had excellent surface finish, the last few years have seen still further developments in this direction.

One section was devoted to a display of American products which use die castings in their construction. These included a food mixer, juice extractor, a parking meter, a domestic scale with the case die cast in zinc alloy, a door check and a lawn mower. There was also an impressive plated Oldsmobile instrument panel, 48 in. long and 7 in. wide at the centre. It may have been national pride, but the writer felt that these exhibits were a little "fussy" and that from the design and die-casting production points of view we in this country could do at least as well.

In the centre of the exhibition was a section illustrating the many finishes which can be applied to zinc alloy die castings. In the enamelled castings the writer noticed red, several shades of blue, greens, cream, orange, brown and grey. The enamels are almost invariably applied after the die casting has been pre-treated with a phosphate dip. Also in this group were plated die castings, mainly in nickel and chromium, but also including a few plated with brass. The well-known Acme wringer was also included in this section illustrating the various finishes which are applied to die castings, and incidentally also showing how a machine of good design and modern appearance can be produced at reasonable cost by the use of die castings.

No one visiting this Exhibition could fail to be stimulated by the progress which the industry is making. Technical queries from a large number of visitors were dealt with by staff of the Zinc Development Association and by members of various die-casting companies.

## Correspondence

### Gas Carburising with Town's Gas

The Editor, METALLURGIA.

Dear Sir,

We were very interested to read Dr. Laidler's account entitled "Gas Carburising with Town's Gas" which appeared in the May issue of METALLURGIA.

During the late war, assisted by Mr. T. S. Lister, we were concerned with a large scale investigation of gas carburising at Messrs. David Brown & Sons (Huddersfield), Ltd. In the course of that work we were privileged to discuss with Dr. J. Taylor the problems of gas carburising and to see reports on the experiments which he and Dr. Laidler were undertaking for the Ministry of Supply and the Iron and Steel Research Council at Kings College, Newcastle-on-Tyne, under the direction of Professor H. L. Riley. These very interesting experiments were concerned with the use of specially treated coal gas as an active carburising medium, and it is hoped that an account of them will be published.

So far as the more fundamental aspects of gas carburising are concerned the results of our own work, also (as yet) unpublished, are in general agreement with the conclusions reached by Riley, Taylor and Laidler, and outlined by Dr. Laidler in the early part of his article. Our opinions differ, however, at the point where consideration is given to the practical application of those fundamental principles to industrial scale gas carburising.

There can be no doubt that if Town's Gas would carburise effectively in its raw condition or without any

extensive pre-treatment, then for many purposes the local Gas Works and even more so a gas grid, would be a very convenient source of supply of carburising gas. On an experimental scale we have in fact used coal gas (containing  $\text{CH}_4$  — 27.5%,  $\text{H}_2$  — 48.0%,  $\text{CO}$  — 6%) successfully as a carburising medium without any previous treatment, but only when the object being carburised was simple in shape and the design of the apparatus such as to ensure a high velocity of gas over all surfaces of the object. Riley, Taylor and Laidler also, we believe, achieved this result.

These ideal conditions rarely obtain with commercial gas carburising, although the carburising of, say, armour plate, might conceivably be considered as an exception. In general, however, the shape of articles to be carburised are complex, and in addition many variations of shape and size render it impracticable to design carburising furnaces which are "tailored" in shape and size to particular components. Hence, as Dr. Laidler indicates, some means must be adopted which will improve the "carburising power" of coal gas so that it can carburise at or near the maximum rate without having recourse to very high rates of flow of gas over the steel surface.

Dr. Laidler chooses an unspecified chemical treatment of coal gas which removes  $\text{O}_2$ ,  $\text{CO}_2$  and  $\text{H}_2\text{O}$  without seriously interfering with the  $\text{CH}_4$  content of the gas. This is a somewhat novel process and our own work confirms that such a gas would have excellent carburising properties if given adequate circulation around the load of work in the carburising furnace. We should be interested to hear whether the gas pre-treatment plant has performed consistently for extended periods under the exigencies of normal works heat treatment shop conditions.

Our own experience over long periods of operation in the heat treatment shop has indicated that auxiliary gas treatment plant is undesirable and that if pretreatment of whatever gas is used could be avoided it would remove one of the most serious disadvantages that has been associated with gas carburising in this country. Any feature which unnecessarily complicates the process subscribes to the almost traditional criticism that gas carburising can only be successful under strict scientific supervision. This aspect was given special consideration in relation to the gas carburising process which we were responsible for developing and which has since been marketed by a well-known furnace manufacturer in this country. It is perhaps appropriate that the claims of this process should be brought to the notice of your readers, particularly from the point of view of its simplicity.

It seemed to us that the most obvious and easy method of improving the "carburising power" of raw coal gas would be the simple addition of a small amount of hydrocarbon gas. From a technical point of view, methane appeared to be the most desirable hydrocarbon, but as it is not commercially available in this country we decided to use butane. The butane was introduced in measured amounts into the coal gas line at a point just before the gas mixture entered the carburising furnace, and, contrary to general opinion, this procedure was an unqualified success. We have no reason to believe that an addition of propane in the correct proportion would not be equally successful.

Much has been said and written about the necessity for the removal of  $\text{CO}_2$ ,  $\text{O}_2$  and  $\text{H}_2\text{O}$ , but this was found to be entirely unnecessary, as indeed might be expected from a consideration of the gas equilibria data. Precautions had of course to be taken in the design of the carburising furnace to ensure that any stabilising reactions occurring in the gas mixture were virtually complete before reaching the surfaces of the components being carburised. An important feature of the furnace was an internal fan which forced a strong positive circulation of the gas through the charge of work, giving consistent and even carburising despite the use of a relatively lean carburising gas. This lean gas, giving an effluent containing as little as 1% methane, did not necessarily carburise, during the initial stage, at the maximum rate referred to by Dr. Laidler, although this condition could be easily achieved, if desired, with an appropriate butane addition. In practice, however, when carburising to depths from 0.020 to 0.060 inch it was found preferable to use a somewhat reduced carburising power, thus increasing the carburising period but avoiding the necessity for a diffusion period. Difficulties due to carbon deposition were quite unknown.

Unlike some gas carburising processes for which complicated gas treatment equipment is claimed to be essential, the simple arrangement which we adopted was used successfully under normal production conditions to carburise many thousands of precision components. No difficulties of either a mechanical or a technical nature were encountered, and the plant could be operated by any reasonably intelligent workman without the constant attention of laboratory staff.

Yours faithfully,

M. L. BECKER, E. INESON.

The British Iron and Steel Research Assn.,  
11, Park Lane, London, W. 1.  
21st May, 1948.

The Editor, METALLURGIA.

Dear Sir,

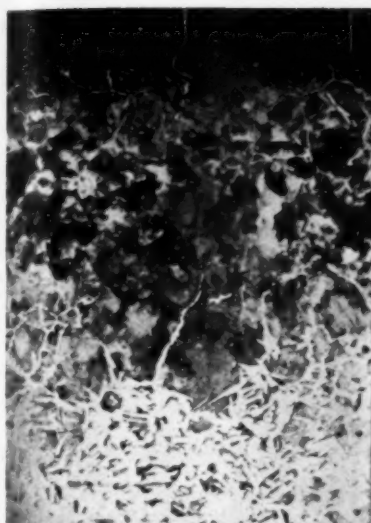
I am most interested and gratified to read the comments of Dr. Becker and Mr. Ineson on the account entitled "Gas Carburising with Town's Gas," particularly as the author has had the privilege of knowing their extensive experiments in this field. In the article, pressure of space prevented any detailed account of the fundamental aspects of gas carburising, and no more than broad outlines of the work which led up to the manufacture of large-scale installations could be given until the researches carried out at Newcastle are published in full. Consequently, the article was only intended as a brief review of the industrial application of the method and its advantages, together with a very limited background of the fundamentals on which the process is based.

It is agreed that the use of raw town's gas leads to some highly interesting and useful results in certain special circumstances, but in most applications it is entirely unsatisfactory, and even in the early days of the war when the country's gas supplies were generally of a much higher standard than obtains at present, it soon became apparent that to achieve an atmosphere which gave complete control over the carburising of those commercial articles which show "so many variations of shape and size" (and in addition, so many variations of material, case depth and type of case required) it was essential to remove or subdue the deleterious constituents  $\text{CO}_2$ ,  $\text{O}_2$ ,  $\text{H}_2\text{O}$  and sulphur. It may be said that with certain town's gas supplies, the effect on the carburising power of the gas of removing them all is even greater than the summation of the improvements effected by removing one at a time. The successful removal of these constituents on an industrial scale is probably as much a physical as a chemical process and is the subject of Wild-Barfield patents Nos. 588, 743 and 588, 547, and we are glad to receive the confirmation of Dr. Becker and Mr. Ineson that their own work shows that such a gas has excellent carburising properties.

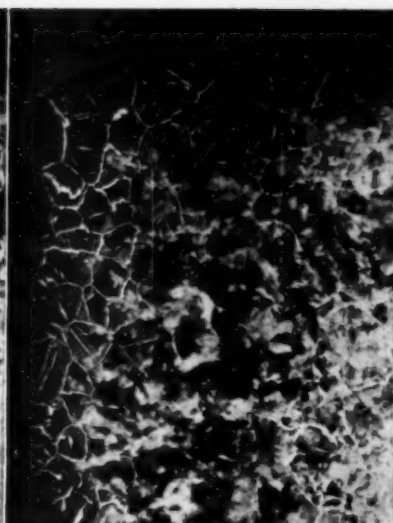
The pre-treatment plant at the Wild-Barfield works has indeed performed successfully for a number of years under the usual works heat-treatment shop conditions, during which time many hundreds of charges have been carried out. Even larger sizes in industry have long given under normal heat-treatment shop conditions a carburising atmosphere which has made possible the precise control of the carburising of some of the finest and most complicated engineering components in the world.

The necessity for strict scientific supervision of any process is a most important point, and the Wild-Barfield plant was also designed with this particularly in mind so that the plant as a whole is operated by ordinary heat-treatment shop personnel. The pre-treatment is automatically carried out by the self-contained preparation unit connected directly to the gas mains and no control other than the exceeding of a certain minimum gas flow for a given furnace size is required.

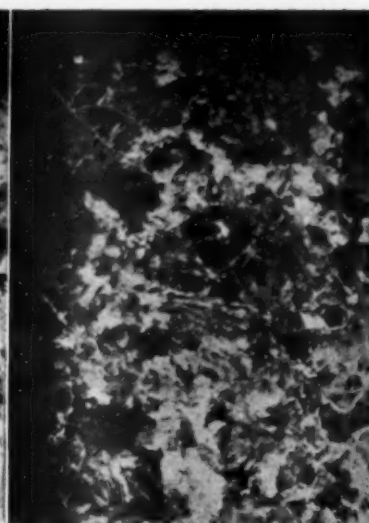
The work of Dr. Becker and Mr. Ineson has shown how the improvement of the carburising power of raw town's gas can be effected by additions of butane, propane, etc., but it is not at all clear how one arrives at the control of the butane/raw town's gas ratio to achieve exactly the right balance to avoid sooting and yet carburise at the maximum rate, bearing in mind



Sample No. 1.—Left, showing 0.020 in. case along straight edge. No diffusion. Note hypereutectoid.  $\times 100$  Etched 2% Picral.



Right, showing case at sharp corner.



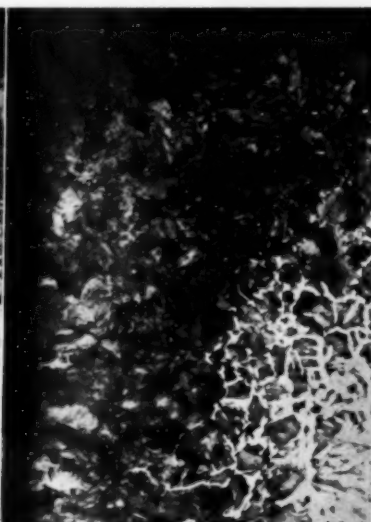
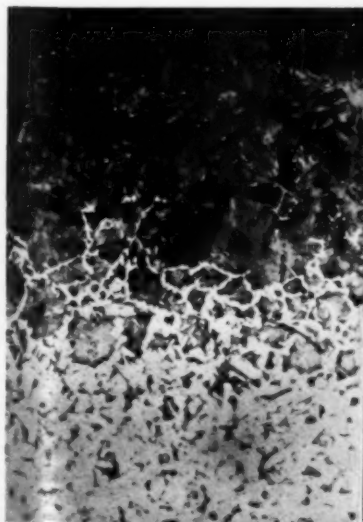
Sample No. 2.—Showing trace of hypereutectoid at sharp corner of sample.  $T/t = 1.25$  (Partially diffused).  $\times 100$ .

the tremendous (and often unknown) variations of "carburising power" required with different charges of widely differing surface areas, steel compositions and the constantly changing requirements as the carburisation proceeds. In fact, the writers appear to confirm the statements that to avoid excessive sooting of an undesirable type it was preferable for them to use a somewhat reduced carburising power and thereby increase the carburising period. The diffusion period should not be regarded as a necessary additional evil, but as a desirable integral part of the total carburising cycle giving rise to a greatly superior type of case in a much shorter time than by using a "lean" gas and

carburising directly to near-eutectoid composition. As a very large proportion of the cost of carburising by any medium lies in the heating of the work and its maintenance at the correct temperature, these factors play a very important part in the economics of industrial processes. It is felt that this is particularly pertinent in the writers' own choice of really deep cases for armour plate, as if one medium carburises twice as rapidly as another the differences between, say, 40 hours and 80 hours at a temperature is much more striking than that between 2 hours and 4 hours for a medium case depth. Again, with certain applications it is possible to carburise at the maximum rate of  $950^{\circ}\text{C}$ ., still avoiding hypereutectoid by simply increasing the proportion of diffusion time.

The statement that the "lean" gas does not necessarily carburise during the initial stage near the maximum rate is particularly important in considering very thin cases, say of the order of 0.015 in./0.020 in., and should a high carbon content be required on such a depth, the appropriate theoretical hydrocarbon addition must be nearing the danger mark for excessive sooting. The photomicrographs (1), (2) and (3) illustrate the control of carbon content of such a thin case using the prepared town's gas to carburise at the maximum rate, and the diffusion principles to obtain great flexibility.

Finally, Dr. Becker and Mr. Ineson stress the importance of an internal fan to introduce circulation of the gas through the charge. It is generally agreed that the use of a fan at high temperatures in a carburising atmosphere introduces the possibilities of mechanical breakdown due to the



Sample No. 3.—Left, showing case along straight edge (fully diffused,  $T/t = 1.50$ ). Right, showing case at sharp corner with complete elimination of free cementite.  $\times 100$ , Etched 2% Picral. Reproduction of these five photomicrographs is slightly reduced from the originals.



severity of the conditions. As pioneers in the use of fans for forced-air circulation, Messrs. Wild-Barfield have spent many years in investigating their use in gas carburising practice. Fundamentally, the efficiency of forced circulation by means of a fan is primarily a function of the density of the gas, and it is generally accepted that forced air circulation furnaces are not manufactured to operate at temperatures in excess of 700/750° C., as the air density is then so low as to render the principle ineffective. It is thus difficult to follow the reasoning which suggests that a gas which has a density relative to air of approx. 0.5 at room temperature can show the well-known characteristics of forced air circulation when the gas is at 925° C. Fans under such conditions do, of course, induce turbulence as distinct from a strong

positive circulation, and it was only after many years of studying the effects and nature of such turbulence that fans were abandoned in favour of suitably designed systems of multiple inlets, also the subject matter of patents, which gave the same turbulent effect without the mechanical difficulties and cost of such fans. Innumerable test and production charges have shown that such systems enable perfect uniformity of case depth and type of case to be obtained even in very large charges weighing  $\frac{3}{4}$  ton.

Yours faithfully,

D. S. LAIDLER.

Wild-Barfield Electric Furnaces, Ltd.,

Watford, Herts.

May 28th, 1948.

### The Late Mr. David Flather

MANY readers who have noted the brief announcement of the death of Mr. David Flather, at the age of 83, will have realised that his death severed an important link with the Sheffield steel industry. He was a life director of Messrs. W. T. Flather, Ltd., the Sheffield steel firm founded by his father. Before joining the family business, in 1886, he was with Messrs. John Brown & Co., Ltd., as their first metallurgical chemist. In 1898 he became a director of W. T. Flather, Ltd.; from 1918 to 1941 he was chairman and in 1946 was made a life director. In addition he had been Master Cutler and was a Sheffield Town Trustee and Church Burgess. Mr. Flather had many interests outside the industrial life of Sheffield. He was a leading figure in Masonic circles a Diocesan lay reader and, since 1928 a City magistrate. He was intensely interested in local history and it is to his interest in this direction that Sheffield owes the memorial to Thomas Bolsover—the inventor of old Sheffield plate—in Whiteley Wood. He defrayed the cost as he believed that "Sheffield and often other cities were not quite as grateful as they should be to those who had laid the foundations for their prosperity."



Mr. Flather took a leading part in the formation of the Sheffield Junior Chamber of Commerce. At the time of his death he was president of the Bright Steel Bar Association and was a former president of the Sheffield Society for the Preservation of Ancient Tools. He was a member of the Chamber of Commerce, of the Committee of the Applied Science Department of the University and of the District Council of the Federation of British

Industries. He was also connected with a number of scientific societies.

At one time Mr. Flather was president of the Sheffield Literary and Philosophical Society and a member of the Hunter Archaeological Society. He was an enthusiastic Mason, commencing in 1888 when he became the first initiate of the Hallamshire Lodge No. 2268. He was a member of that Lodge for 60 years. He received high provincial rank and Grand rank, and was an authority on the history and traditions of Freemasonry.

Mr. Flather left two sons, Mr. Geoffrey M. Flather, joint Managing Director of the firm, and Major K. D. Flather, a director, and one daughter, Mrs. P. M. Hoff, now living in America.

### New Aluminium Research Laboratory

PLANS for the construction at Arvida, Quebec, of a large new research laboratory for more extensive investigation of problems in the production of aluminium have been announced by officials of Aluminium Laboratories Limited, subsidiary of Aluminium Limited. The new laboratory, with which will be incorporated a pilot production plant, is expected to be completed in 1949. The building and equipment, costing in the vicinity of one million dollars, will provide augmented facilities for an already large staff of research scientists and technicians.

Research on aluminium has been proceeding at Arvida for a considerable time. This city is a natural choice for the location and extension of research facilities since it is the site of the large aluminium production plant operated by Aluminium Company of Canada, Ltd. The latter company is the principal user of the results of the research.

The Arvida laboratory will be the third modern unit operated by Aluminium Laboratories Limited for industrial research for the Aluminium Limited group of companies. The other laboratories, one at Kingston, Ontario, and the second at Banbury, England, are also adjacent to operating plants and are largely concerned with problems in the fabrication of semi-finished and finished products from light metals. The research at Arvida, on the other hand, is concentrated on problems in the actual production of light metals, up to and including the extraction of metallic aluminium and magnesium. This involves investigation of the processes used for the chemical treatment of basic ores and other raw materials and for the electro-chemical and electro-thermal reduction of the metals from their oxides.

# The Iron and Steel Institute

## Annual General Meeting in London

*The annual meeting of this Institute proved to be very successful socially and technically. It was well supported by members and, with many overseas members and visitors present, it had, in some respects, a pre-war character. Brief reference is made to the initial meeting, the President's Address is summarised and some of the main aspects are given of the papers presented at the technical sessions and of the discussions which followed.*

**T**HE Seventy-ninth Annual General Meeting of the Iron and Steel Institute was held at the Royal Institution of Chartered Surveyors, in London, on 5th and 6th May, 1948. The retiring President, Dr. C. H. Desch, F.R.S., in opening the meeting, referred to the loss the Institute had sustained by the death of Mr. Benjamin Talbot, a Past-President and Bessemer Medallist of the Institute.

### Welcome to Overseas Visitors

The President then welcomed visitors from overseas, among whom were Dr. R. F. Mehl and Mr. F. D. Foote (U.S.A.), Dr. Durrer, M. Zehnder and M. Hubscher (Switzerland), Dr. Goerrissen and Mr. Schjelderup (Norway), Mr. Bo. Tigerschiöld and Mr. Bonthron (Sweden), Mr. R. de Quijano and Mr. L. Barreiro (Spain), Prof. Lefebvre (Belgium) and M. Rocquet (France).

### Report of Council

In presenting the Report of Council for 1947, Dr. Desch referred to the successful Summer Meeting held in Switzerland last year and said that it was hoped to accept an invitation to visit Norway next summer. The year's publications of the Institute included a Symposium on Hardenability and one on Powder Metallurgy and a report on "Qualifications for Entrance to University Schools of Metallurgy," had been published by the Joint Committee on Metallurgical Education.

### Award of the Bessemer Gold Medal

The President, in presenting the Bessemer Gold Medal for 1948 to Mr. W. J. Dawson, C.B.E. (Hon. Vice-President), said the award was in recognition of his contributions to the development of steel castings and to the production of alloy and heat-resisting steels. Mr. Dawson had been connected with the steel industry for over fifty years and had taken a leading part in co-operative research, serving on numerous technical committees. In recognition of his work as chairman of the Steel Castings Research Committee, he had been awarded the E. J. Fox Gold Medal of the Institute of British Foundrymen.

Mr. Dawson, in reply, thanked the Council for the award and described it as a very rich and ample reward for what he had been able to do for the industry during the last fifty years.

### Other Awards

The Sir Robert Hadfield Medal was presented to Mr. A. PREECE in recognition of his work on the overheating and burning of steel. Mr. R. FOWLER was the recipient of the Williams' Prize for his paper on "Blowing-Out a Blast Furnace." An Andrew Carnegie Travelling Scholarship had been awarded to Mr. A. W. POSTLETHWAITE for the study of powder metallurgy in the U.S.A.

### Changes on the Council

*New Member of Council:* Mr. T. Jolley.

### New Honorary Members of Council:

Sir Arthur Smout, President of the Institute of Metals.

Dr. C. Sykes, F.R.S., President of the Sheffield Metallurgical Association.

Mr. Basil W. Doncaster, President of the Sheffield Society of Engineers and Metallurgists.

Mr. H. F. Padbury, President of the Ebbw Vale Metallurgical Society.

Mr. A. Preece, President of the Leeds Metallurgical Society.

All retiring Vice-Presidents and Members of Council were re-elected.

### Induction of Sir Andrew McCance, D.Sc., F.R.S.

Dr. Desch said that the steel industry had a number of distinguished industrialists and many distinguished research workers, but the combination of the two in one man, as we had in Sir Andrew McCance was very rare. It was still more remarkable that, sharing the direction of a great business, he had still found it possible to carry on research work of an abstruse character. Sir Andrew first entered the steel industry with Messrs. Wm. Beardmore & Co., later becoming Managing Director of the Clyde Alloy Steel Co. At this time he acted as adviser to Messrs. David Colville & Sons, Ltd., joining the Board in 1930. Since then he had always been associated with Colvilles and had taken a leading part in every development of the company.

He became a Fellow of the Royal Society in 1943 and was first chairman of the British Iron and Steel Research Association. In 1914 he became a member of the Institute, a Member of Council in 1932 and a Vice-President in 1941. In 1940 he was awarded the Bessemer Gold Medal and in 1947 the honour of knighthood was conferred on him by the King.

He was the author of many technical papers and had been particularly active in the field of physical chemistry as applied to steelmaking.

As one who was held in the very highest respect by all the members it gave him very great pleasure to induct Sir Andrew as President of the Institute.

Sir Andrew McCance then took the chair and as his first duty proposed that they should express their thanks to Dr. Desch for his services as President. This was seconded by Sir Peter Brown and carried with acclamation. Dr. Desch suitably responded.

Before delivering his presidential address, Sir Andrew McCance expressed his appreciation of the honour conferred upon him by his election as President.

### PRESIDENTIAL ADDRESS

#### The Development of the Open Hearth Furnace

Whilst there have been no basic changes in the methods of steelmaking since the inventions of Bessemer and Siemens, considerable progress has been made in productive efficiency. It was not until

the advent of the railway age that the limitations of the crucible process were appreciated. The alternative idea of making steel in a furnace hearth occurred to many people, but it remained for a man not primarily interested in steel manufacture to open up the path of progress. Siemens was very interested in the scientific developments of his time, and the problem of heat conservation was constantly in his mind. Based on an idea of Stirling, he introduced the regenerative principle into the design of a billet heating furnace, but it was not until his invention of the gas producer and his second patent of 1861, that the regenerative furnace as we know it came into being. The first attempts at steelmaking by the oxidation of pig-iron by iron ore were unsuccessful, but the process was made a practical proposition by Martin, who used a silica sand hearth and diluted the carbon in the pig-iron by the addition of wrought-iron scrap before he attempted to remove it by ore.

Furnace capacities rose in a few years to 10-15 tons, but there was then a pause in the rate of growth, occasioned no doubt by a desire to gain experience both in engineering construction and in metallurgical control. The introduction of mechanical charging increased the output by about 10% and enabled larger furnaces to be built. By the beginning of the century furnaces of 30-40 tons capacity were in operation, but the gain in output was somewhat doubtful as the larger furnaces were less thermally efficient and considerably fewer charges per week were obtained. This state of affairs naturally stimulated investigation and invention in an endeavour to improve thermal efficiency, but progress was slow and by 1910, 70% of British furnaces were still of 40 tons capacity or less. It was not until after 1918 that the benefits from the various technical improvements were realised, for by that time economic considerations had made intensive production a necessity. From that time furnaces have gradually increased in size and the output for a given size of furnace has also been improved.

The design of melting furnaces is still entirely empirical but a consideration of the factors involved shows that for an output proportional to the size of furnace, there are certain relationships between the hearth area, bath depth and capacity of the furnace.

It is general experience that the time required to work the charge increases with increase in charging time, so that every effort should be made to reduce the latter. Such points as size of charging boxes, position of full boxes relative to the furnace, speed of cranes and chargers etc., are worthy of consideration in order to ensure that no time is wasted. Larger furnaces should have larger charging boxes, which in turn means larger doors and water cooled arches. By a study of these factors, a charging rate of 40-50% of the capacity per hour should be capable of achievement.

#### Operating Factors

In the charging and melting period, melting is facilitated by a hot intense flame directed down through the charge and a large difference between gas and air velocities is required to achieve this. The Venturi design is probably the best attempt, with velocity differences of 100-150 ft./sec., although other furnaces are in operation with only 30-40% of this figure. When the charge is melted, radiation from the flame is secondary to that from the walls and roof, and the latter is limited by the refractories. If the use of basic bricks would enable the walls and roof to run at 100° C. higher in temperature,

the rate of heat input to the bath from the flame could be raised to 36% instead of the present 11% with consequent faster working.

Slag composition requirements are such that it does not seem likely that alterations can be made to improve production, but the problem of increasing the slag-metal interface still awaits a solution. The absorption of heat when carbon is oxidised by FeO is avoided when direct oxidation is carried out and there is great scope for the use of oxygen in the open-hearth furnace. It is a great help in shortening the time taken to reduce the carbon to a low figure, say 0.04-0.08%, but at these low values so much is required to increase the FeO in the slag that its efficiency is low. A pronounced increase in refractory consumption occurs as a result of splashing of slag on the walls and roof due to the rapid evolution of carbon monoxide.

With high production furnaces, the organisation of casting and handling the ingots requires careful consideration. Even car casting with a separate bay requires the ingot size to be related to the furnace capacity and a figure of 15-20 ingots per cast seems to be the ideal.

Even in a well designed furnace, the attainment of a high production rate entails some extravagance in the use of fuel and, although a large amount can be recovered in waste heat boilers, a serious attempt should be made to recover the remainder. One of the main obstacles is, of course, the corrosive effect of the SO<sub>2</sub> in the gases when the temperature falls below the condensation range.

It is only by a determination to find a use for all the products which have hitherto been regarded as waste, as well as by a correct understanding of the factors concerned in intensive production that the economic success of the steel industry can be maintained.

#### W. H. Hatfield Memorial Lecture

In the evening Professor Robert F. Mehl, of the Carnegie Institute of Technology, Pittsburgh, delivered the Third Hatfield Memorial Lecture to a large and appreciative audience in the Lecture Hall of the Royal Institution of Chartered Surveyors. Dr. Mehl's subject was, "The Decomposition of Austenite by Nucleation and Growth Processes."

#### Technical Sessions

Following the Presidential Address the remainder of the meeting was devoted to the presentation and discussion of technical papers. In all, thirteen papers were presented, and in the following notes we have endeavoured to give a brief digest of the papers and some of the points which arose in the lively discussion which followed.

#### CONSTRUCTION AND REPAIR OF OPEN-HEARTH FURNACES.

By D. C. MUIR.

THE most important factor governing the output of steel from a particular furnace is its availability, i.e., the percentage of time in which it is capable of making steel. It is obviously possible to improve the availability in two ways, by increasing the furnace life and by reducing repair and rebuilding times. The paper discusses the means employed at the works of the Consett Iron Co., Ltd., to effect improvements in these two ways. An account is given of the furnace repair programme over a period of seven years and of a system of balanced repairs whereby nine out of ten furnaces are available at any one time. The furnaces concerned were



seven 75-ton basic furnaces, and two 75-ton and one 40-ton acid furnaces, all originally designed for cold pig and scrap. Since 1941, the basic furnaces have used hot metal instead of cold pig. The campaign of a basic furnace was originally fixed at 30 weeks (with a shut down for top dressing after 15 weeks), and that of an acid furnace at 24 weeks, but wartime operating conditions resulted in shorter lives, particularly for the basic furnaces. Failure of the roof and the choking of the checkers and flues limit the life of the basic furnaces, whilst in the acid furnaces the filling up of the slag pockets is the governing factor. In order to minimise the time taken in rebuilding, it is essential to plan the flow of labour and materials, and in this way a rebuild can be effected in 12-15 days, according to the repair work necessary in the slag chamber and regenerator arches.

The following points are mentioned concerning increased availability: (1) The roof life is increased by careful glazing during the first few weeks, as a result of increased conductivity. (2) The use of mixtures based on stabilized dolomite for tap-hole formation and hearth repairs has reduced both the dolomite consumption and the time lost due to bad hearths. (3) The top layers of basic furnace checkers last longer when high porosity semi-silica bricks are used, due to their ability to absorb slag to some depth without melting or distortion. (4) In the semi-sloping back walls, chrome-magnesite bricks should be used up to such a height that the flame attack is on them rather than on the silica bricks. (5) Experience with basic linings to the air uptakes has reduced the repairs necessary and it is proposed to try a chrome-magnesite lining on the gas uptakes. (6) Special silica blocks are used for the gas ports, which are scotch blocked after 9-10 weeks. Chrome-magnesite blocks were not successful, as the present design is not sufficiently stable for a basic brick structure, which lacks the mechanical strength of a silica brick structure in which the bricks become fluxed together. (7) The time taken to empty slag pockets can be reduced by building a false wall inside the pocket and filling the 3-inch gap between it and the wall proper with coke breeze. (8) By the use of a relieving arch across the uptakes, it has been found possible to build the end walls above stage level, without waiting for the completion of the repairs to the slag chamber.

For the future, it is hoped that the introduction of all-basic furnaces will increase the furnace life and the rate of production by permitting the use of higher temperatures. It is foreseen that it may be difficult to increase correspondingly the life of the slag pockets and checkers, but there is reason to believe that the use of liquid fuel and/or coke-oven gas will help to this end. It is also intended to insulate the above-stage sections to conserve fuel and this will depend on the development of a basic hot-face insulating brick.

#### THE DESIGN OF OPEN-HEARTH GAS PORTS.

By M. P. NEWBY, B.Sc., A.Inst.P.

A STUDY is being made by BISRA of the various factors influencing the mixing of gas and air streams in the open-hearth furnace, using aerodynamic models for the experimental work, and the experiments described in the paper were made with a view to determining the efficiency of a port considered as an arrangement for converting pressure energy into kinetic energy of gas motion. This is an important function of the port, as it has been shown previously that combustion in the

flame is dependent on the intimate mixing of the gas and air, and that an increased rate of mixing can be achieved by increasing the velocity of the gas and air streams, and by increasing the difference in velocity between the two. Rapidity of mixing is advantageous in that it ensures a high flame temperature early in the passage of the gases through the furnace chamber.

After an outline of the theoretical considerations involved, Mr. Newby describes the experimental procedure. The port design of a particular furnace was chosen for the work, and scale models were made of the gas uptake and port. An improvement of some 4% could be made, in entry velocity, by various modifications to the original parallel sided port. A simple Venturi slope results in increased resistance to the outgoing gases, but the incorporation of a parallel portion at the throat reduces the outgoing resistance to that of the parallel port, without reducing the beneficial effect of the Venturi on the ingoing gas. The addition of a 90° trumpet between the port and the furnace greatly reduced the outgoing resistance without affecting the flow into the furnace. This is considered impracticable as, unless very effective water cooling could be used, the gases would smooth off the brickwork and the velocity into the furnace would be reduced by a recovery of static head in the throat.

It is concluded that any appreciable increase in velocity will necessitate an increase in pressure across the port, which may present practical difficulties.

#### INSTALLATION AND USE OF INSTRUMENTS ON OPEN-HEARTH MELTING FURNACES.

By R. C. BAKER, A.Met., A.I.M.

THE application of instrumentation to a 50-ton acid furnace and a 40-ton basic furnace is described. At present the following are in use: (1) Automatic draught control using regulated cold air leakage into the bottom of the stack. The controller is a Hopkinson-Carlstedt regulator and the control point is 18 in. above sill level on the front wall of the furnace. (2) Producer gas and air flow indicators and recorders. (3) Gas and air checkers waste-gas temperature recorders. (4) Furnace roof temperature indicators and recorders. (5) Immersion pyrometers for liquid steel temperature measurement. These are available for use by the furnacemen whenever required. The temperature before tapping is taken for record purposes.

A feature of the installation is the recording of crown temperature, gas and air flow on one chart of a six point potentiometric recorder.

The crown temperature is measured by Fery radiation pyrometers and attempts are being made at control by controlling the gas regulating valve. This is satisfactory for normal working but it cannot yet deal with an empty furnace, a furnace ready for tapping, nor a furnace with a door open for a considerable period, but attempts are being made to overcome these difficulties. It has been found that furnacemen appreciate and use the instruments and attempt to maintain the crown at a predetermined limiting temperature, with the result that melting rates have been substantially increased.

#### Joint Discussion

DR. J. H. CHESTERS (United Steel Companies, Ltd.) referring to the all-basic furnace urged users to record all possible information on the running of these furnaces. A schedule of information required would be issued shortly by the All-

Basic Furnace Sub-Committee. He saw advantages in the porous basic insulating brick in reduced raw material costs, lower conductivity and lower bursting coefficients, but against these he pointed out their weakness, lesser resistance to slag attack and the difficulty of making them.

Turning to Mr. Newby's paper, Dr. Chesters said that in experiments with a water model of the same port he had found marked recirculation of the gas. He was sorry that Mr. Newby considered that increased gas velocities could only be achieved by higher pressures, as this would result in difficulties in the gas producer plant and in increased leakage in the furnace. He asked whether Mr. Newby had investigated the effect of surface roughness in his models.

MR. F. L. ROBERTSON (John Summers & Sons, Ltd.) referring to Mr. Baker's use of multiple charts, suggested an extension of them to give a complete record of the heat, including the charge, ore feed, etc., and the carbon and sulphur figures as reported during the refining.

DR. A. H. LECKIE (B.I.S.R.A.) questioned whether the method of automatic pressure control, by means of air leak to the stack, would not spoil the waste heat boiler performance. With regard to temperature control, apart from the abnormal conditions mentioned by Mr. Baker, would a simple proportional control be sufficient or would it be necessary for instrument makers to put in derivatives.

He was not sure whether the friction factors in Mr. Newby's paper included the effect of loss of velocity head in going into the furnace. If not the figures were reasonable, otherwise they seemed very low. There were other methods of getting good mixing, such as by the use of smaller ports, and by making the gas and air meet at right angles.

DR. D. F. MARSHALL (Park Gate Iron and Steel Co., Ltd.) suggested that by the use of instrumentation, standardised furnace working could be established. It would then be an easier matter to forecast furnace life and draw up a planned repair programme.

MR. R. E. H. WILLIAMS (Wellman Smith Owen Engineering Co., Ltd.) commented on the higher figures given by Mr. Muir for silica brick and cement consumption and for man-hours expended in furnace rebuilding, as compared with those given recently by Mr. Kilby. Whilst appreciating the difficulties of comparison between one plant and another he thought that the waste-gas velocity was a major factor in the destruction of refractories.

MR. M. W. THRING (B.I.S.R.A.) pointed out the complementary nature of the air and water models, the former being ideal for measuring pressures and factors of mixing whilst the latter shows flow patterns very effectively. All new port designs should be tested by air models, as Mr. Newby had now devised a means of measuring factors of mixing, and the experiments were showing that more could be done with the air port than the gas port. Now that mixing factors could be measured, it would be worth while measuring the factor for the port with the 90° cone after smoothing out, in order to see whether it gave better results than had been anticipated when only the gas port had been considered. It should be possible to carry out model mixing experiments for oil furnaces using a heavy gas as the oil.

MR. H. PARNHAM (General Refractories, Ltd.) asked whether Mr. Muir considered that metal plate joints were necessary between chemically bonded bricks in the back wall, and whether the extra cost involved was offset by an increase in life. He also asked whether Mr. Muir preferred a chrome-magnesite unfired brick to a magnesite-chrome brick from the point of view of life.

## Reply to Discussion

MR. D. C. MUIR said that instrumentation had not been mentioned because he had insufficient data to correlate with furnace life. Considerable wear had occurred on furnace ends but he thought that other factors than waste-gas velocity were concerned in the wear of checkers. The use of metal plate joints in the back wall was justified by an increase of 2-3 weeks in the life, but as the plates were a nuisance to the bricklayers, attempts were being made to introduce the metal in some kind of cement. He preferred magnesite-chrome bricks for the roof and chrome-magnesite for the back lining.

MR. M. P. NEWBY said that he had not tried the effect of surface roughness in his models, but the results were comparable between one model and another and he thought that the effect would be a small fraction of the friction factor of the actual port, as shown by the comparison of furnace trials with model results. The fact that friction factors were lower than expected, demonstrated the superiority of model tests over guesses from data gathered from conventional bends. He agreed with Dr. Leckie that other methods of attaining improved mixing were possible and they were now working on those lines.

MR. R. C. BAKER agreed that with waste heat boilers, pressure control by stack air leakage was unsuitable and in such cases the butterfly damper of the boiler was used. With regard to automatic temperature control he had no experience of proportional control by itself, as the control used was floating with the first derivative function added later.

## THE PRODUCTION OF NODULAR GRAPHITE STRUCTURES IN CAST IRON.

By H. MORROGH AND W. J. WILLIAMS, B.Sc.

CONTINUING their work on the formation of spherulitic graphite in nickel-carbon and cobalt-carbon alloys, the authors have, by way of the iron-nickel-carbon and iron-carbon alloys, attained their objective of producing a cast iron with nodular graphite in the as-cast state. This, of course, is an outstanding achievement in the development of cast iron as it means that very good mechanical properties may be attained without prolonged heat-treatment.

The addition of calcium and magnesium to cast iron and to iron-rich iron-nickel-carbon alloys did not result in the formation of nodular graphite, as had been the case with the nickel-carbon and cobalt-carbon alloys, but extensive trials resulted in the discovery that cerium, added to the molten alloys in the hyper-eutectic range, resulted in the formation of nodular graphite on solidification, even with very low nickel contents. The composition requirements of the iron are as follows:—(1) Untreated, the iron must solidify grey. (2) With 0-10% nickel, the iron should be hypereutectic. (3) Silicon should be 2.3-7%. With the 10-15% silicon irons, only the hypereutectic graphite can be obtained with certainty in the nodular form. (4) 3% of nickel and/or copper can be considered equivalent to 1% of silicon. (5) The manganese content may have any value provided the other conditions are met. (6) The sulphur content of the metal to which the cerium is added should not exceed 0.06%. (7) Phosphorus should not exceed 0.5% and should preferably be below 0.1%. (8) Copper and nickel may be present in any amounts. (9) Chromium molybdenum and vanadium may be present in any amounts, provided the other conditions are observed. (10) Titanium in the amounts present in British pig irons exerts no effect.

Cerium-treated hyper-eutectic irons have a structure containing spherulites and "quasi-flake graphite," the proportion of the latter decreasing with increasing cerium. This results in increased mechanical properties and examples are given of hyper-eutectic unalloyed irons and an austenitic iron. The cerium is added to the molten iron as *mischmetal*, which contains roughly 50% of cerium and 50% of other rare earths together with impurities. On remelting the cerium is lost and a reversion to normal flake graphite occurs.

Cerium is a desulphuriser and a powerful carbide stabiliser, and one of its functions in producing nodular cast iron is the reduction of sulphur to a low value. It is considered that the hyper-eutectic graphite forms in the liquid with a spherulitic structure, and that the "quasi-flake graphite" results from the decomposition of a white-iron eutectic after solidification. No "quasi-flake graphite" forms in the vicinity of the spherulites and it is thought that the eutectic graphite in these areas forms on the existing spherulites. Increase of the number of spherulites, by addition of a graphitising inoculant after the cerium addition, results in an iron with the whole of the graphite in the nodular form. The mechanical properties of such alloys are extremely good and several examples are quoted with tensile strengths of the order of 30-35 tons per sq. in.

Although successful trials have been made on an industrial scale, it is too early to assess the commercial possibilities of the process, and it is hoped to present further papers shortly dealing with the practical issues involved in its application.

#### THE CARBIDES IN IRON-CARBON-SILICON ALLOYS AND CAST IRONS.

By D. MARLES.

FOLLOWING up the suggestion, made by Stead in 1910, that iron-carbon silicon alloys contain an iron silico-carbide which is distinguishable from cementite by heat-tinting, the author has studied the carbides in a series of iron-carbon-silicon alloys, with carbon contents of 1.7-3.6%, and silicon contents up to 7%, in order to determine whether Stead's silico-carbide occurs in cast iron of low phosphorus, manganese and sulphur contents, and what limits of silicon are necessary for its formation. By studying the alloys metallographically, the author concludes that, under his particular casting conditions, the carbide phase is duplex for silicon contents between 2.5% and 7%, the amount of silico-carbide increasing with silicon, until at 7% silicon there is no cementite left in the carbide phase. Experiments on the annealing of an iron containing the duplex phase, show that the silico-carbide is less stable than the cementite.

#### Joint Discussion

DR. L. B. PFEIL, O.B.E. (The Mond Nickel Co., Ltd.) said the International Nickel Co. had been investigating the nodular cast iron problem and had found that magnesium, added as a nickel magnesium alloy had given results similar to those obtained with cerium. He stressed the need for work to proceed on the fundamental aspects as well as on the task of converting the authors' findings into an economic proposition.

MR. W. S. OWEN (Liverpool University) was disturbed to find that Mr. Marles had found it necessary to introduce a new phase to interpret the micro-structure of his alloys. This would involve changes in the ternary diagram which were not supported by previous work. He considered that con-

siderable segregation of dissolved silicon could be expected in the carbide phase, and that under certain conditions of metallographic preparation there might be the appearance of two phases. Possibly the number of phases could be determined by X-rays. Along with his colleagues, Mr. Owen said he was carrying out extensive thermal and X-ray investigations of high purity alloys of this type.

MR. M. M. HALLETT (Sheepbridge Stokes Centrifugal Castings Co., Ltd.) emphasised Mr. Williams' introductory remark that quite good mechanical properties could be obtained with the "quasi-flake graphite" structure. Comparing sand and centrifugal castings, he had found that less cerium was necessary for a given structure with the centrifugal castings, presumably due to the increased cooling rate. In the case of his centrifugal castings, the tensile strength peak occurred whilst the graphite was still in the "quasi-flake" form, although the matter is complicated by the occurrence of carbide in the higher cerium alloys.

MR. H. K. WRIGHT (British Rollmakers Corporation, Ltd.) stressed the importance of the fundamental work on graphite formation and hoped that it would ultimately lead to an understanding of the many apparent anomalies encountered in dealing with cast iron. Sulphur seemed to be an important element and he wondered whether in its elemental form or as a sulphide it could dissolve in iron carbide, to a limited extent, and influence the rate of decomposition of the carbide. Was it possible that the suggestion of Baukloh and Meierling, that cerium forms  $CeC_3$  which decomposes exothermally into  $CeC_2 + C$  was correct? The liberated carbon might then act as nuclei for spherulitic growth.

MR. C. H. PAYNE (Qualcast, Ltd.) said the authors had referred to the fact that nodular irons showed inverse chilling in thin sections in the same way as did undercooled graphite irons, and considered it to be due to pressure upsetting normal graphitisation. Whether or not this was the case, there was evidence of an appreciable pressure of graphitisation in both nodular and undercooled irons. There was also some evidence that in the absence of an inverse chill, internal unsoundness might occur where the chill would have been expected. This might be a gas phenomenon, the chill occurring if the gas remained in combination and unsoundness if the gas were liberated. He had experienced a case where an increase in phosphorus had removed the tendency for inverse chilling to occur, and had attributed the fact to the effect of phosphorus in widening the solidification range. Might not phosphorus act in this way to inhibit cerium treatment, or had the authors evidence that there was some combination between cerium and phosphorus. Some years ago he had found the duplex carbide structure referred to by Mr. Marles, on heat-tinting a region of inverse chill.

MR. R. J. BROWN (Morris Motors, Ltd.) observed that mass effect played an important part in the formation of nodular graphite and that variations in graphite formation must be expected in different parts of a casting. For a given melt of cerium-treated iron there was an optimum cooling rate for the production of nodular graphite. Lower cooling rates could give "quasi-flake graphite" and still lower rates undercooled graphite. The authors were probably correct in suggesting that cerium introduced nuclei into the melt. Possibly the "quasi-flake graphite" was formed by the deposition of eutectic graphite on the initially-formed undercooled hyper-eutectic graphite.

DR. J. H. WHITELEY (Consett Iron Co., Ltd.) was pleased to see reference made to Dr. Stead's pioneer work. He



suggested that the system concerned in the duplex structure was a eutectic of iron carbide and silico-carbide with limited solid solubility in each other.

### Reply to Discussion

MR. W. J. WILLIAMS said he was particularly interested in Dr. Pfeil's remarks. If the mechanism were the same with magnesium as with cerium, he wondered whether any carbides were formed with excess of magnesium and whether a white iron resulted. Many of the points raised concerned the mechanism of the graphite formation which would probably be the subject of another paper. He would answer points in more detail in writing but he would point out, in connection with Mr. Hallett's remarks, that with the iron recommended for cerium treatment, "quasi-flake graphite" could be produced merely by rapid cooling of small specimens immediately solidification was complete.

MR. D. MARLES said he would look forward to the publication of the results of Mr. Owen's work. X-ray examination of carbides extracted by cold 10% hydrochloric acid had not been very enlightening, probably as a result of some decomposition taking place in the higher silicon-levels.

### ABNORMAL CREEP IN CARBON STEELS.

By J. GLEN, B.Sc., A.R.T.C., A.I.M.

THE use of large amounts of aluminium in the deoxidation of steel results in abnormally poor creep properties. Short time creep tests have been carried out on low carbon steels with 0.4-1.5% Mn, 0.01-0.15% Si, and 0.0-1.1% Mo, with varying amounts of aluminium up to 3 lbs./ton, the ferritic grain size effect being minimised by normalising at 920° C. Testing was carried out at 8 tons/sq. in. and 450° C. for 5 days, conditions which have been shown to indicate whether a steel has abnormal creep properties. The abnormal creep resulting from aluminium additions is reduced by manganese, silicon and molybdenum in the range examined. All the steels with high creep rate were fine grained but in the higher ranges of manganese, silicon and molybdenum examined, although the steels were fine grained, the creep rate was not excessive unless excess aluminium was present. The abnormality is only marked in low carbon steels, when the aluminium addition is sufficient to give the steel a fine McQuaid-Ehn grain size, so that aluminium may be used in deoxidising provided the grain size remains coarse. The graphitisation effect of aluminium is also confined to fine grained steels. A serious limitation of the short-time creep test is its inability to introduce the effects of prolonged exposure at temperature. Silicon is shown to improve the creep resistance, but it is well known that it promotes spheroidisation and graphitisation which in long term tests may prove harmful.

### THE CREEP PROPERTIES OF MOLYBDENUM, CHROME-MOLYBDENUM AND MOLYBDENUM-VANADIUM STEELS.

By J. GLEN, B.Sc., A.R.T.C., A.I.M.

THE author enumerates the important variables to be considered in the study of creep, viz.: (1) Testing conditions—Temperature, stress, time and atmosphere, and (2) Type of steel—Composition, method of manufacture and deoxidation, fabrication, heat-treatment and microstructure. It is important that temperature, stress and time should be considered together as shown by the fact that the relative creep resistances of two steels may be reversed by changing the stress at the same temperature. Creep tests have been carried out at tem-

peratures of 550° C. and 600° C., and stresses of 4, 6 and 9 tons/sq. in. on 0.5% Mo steel, 0.8% Cr-0.5% Mo steel and 0.5% Mo-0.25% V steels. Aluminium, and to a lesser extent silicon, is shown to be detrimental to the long-time creep properties of molybdenum steel and chrome-molybdenum steel, except at low working temperatures, whilst within the limits considered, manganese, chromium and vanadium, especially the latter, improve them. Care is needed, however, in assessing the effect of composition as it is so dependent on heat treatment. For example, increasing the normalising temperature increases the long-time creep properties at higher temperatures and decreases them at lower temperatures. As a higher normalising temperature is necessary for electric furnace steels to give the same properties as open-hearth steels, the latter are to be preferred for use at high temperatures. The effect of tempering prior to testing has been shown to be an initial stiffening effect followed by a deterioration as tempering time increases. With chrome-molybdenum steel, the stiffening is more pronounced than, and the deterioration not so marked as with molybdenum steel, whilst with molybdenum-vanadium steel the stiffening continues for a long time at normal working temperatures and deterioration will not occur in the normal life of the steel. Indeed molybdenum-vanadium steel should be tempered before service to obtain the full advantage of the stiffening effect. Tempering of this steel also results in greater ductility before fracture and a higher room temperature impact value provided the tempering time is not too short.

Long-time rupture tests are described, and it is shown that molybdenum steel is prone to fail with a low elongation, whilst chrome-molybdenum steel fails with good ductility although the fracture is still intercrystalline. Normalised and tempered molybdenum-vanadium steel shows good ductility, but in the normalised condition it is even worse than molybdenum steel.

Attention is drawn to the limitations of short-time tests when estimating the creep properties of a new steel. In attempting to determine long-time creep properties, it is essential that the tests, particularly those at low temperatures, be carried on for a sufficiently long time to define the shape of temperature-time curves of iso-strain, otherwise there is a possibility of the creep properties being under-estimated.

Once the properties of a type of steel are established, it is necessary to devise a short-time test to check the quality of subsequent supplies. The results demanded should not be unduly stringent as it is difficult to obtain exactly similar results from cast to cast of the same steel, but most of the variations occur in the initial stages of test and there should be no marked variation from the slope of the standard curve once the initial stages are past.

### Joint Discussion

MR. W. E. BARDGETT (United Steel Companies, Ltd.) said that his own work confirmed the author's with regard to aluminium, but indicated that possibly higher values were required to affect the creep rate markedly. Generally speaking, a coarse grained steel would not show abnormality, but a fine grained steel might or might not. In the absence of a range of stresses and temperatures which would permit extrapolations to working stresses and temperatures, the results must be considered as applying to the particular test conditions. It would have been especially interesting if the effect of tempering had been examined at working temperatures and stresses. In contrast to the author's statement that furnace cooling is detrimental to creep resistance, Mr.

Bardgett gave some results which showed that a chrome-molybdenum steel had better creep resistance in the annealed condition than in the normalised condition when tested under working conditions. The statement that open-hearth steels were superior to electric steels was open to question. He presented results to show that under working conditions there was no indication that open-hearth steels were superior, and pointed out that the deterioration evident at higher temperatures might be insignificant under those conditions. The phosphorus content of the open-hearth steels tested was appreciably higher than in the electric steels and the manganese was slightly higher. In view of his own findings with regard to the beneficial effects of phosphorus and Mr. Glen's results on the effect of manganese, he did not think the steels were strictly comparable.

DR. C. H. M. JENKINS (National Physical Laboratory) said that it appeared from the second paper that the author took a value of 50 millionths/hour as being the transition between normal and abnormal whereas he would suggest a figure such as 2 or 10. The oxygen content of some steels tested at the N.P.L. was greatly reduced by the aluminium addition and it was found that low oxygen contents corresponded with high creep rates. The question whether a steel was open-hearth or electric was one of oxygen content.

DR. R. W. BAILEY (Metropolitan-Vickers Electrical Co., Ltd.) said that providing abnormality could be avoided, there was a good field for the further use of carbon steels. Just as Mr. Glen had pointed out there was a range of application with temperature for one carbon content and another, so with the heat-treatment of the steel. Molybdenum-vanadium steel was also being used hardened and tempered, in which condition it was more effective at lower temperatures. In this connection it should be borne in mind that the title of the paper was too wide, as only normalised and tempered steels were concerned. The point had been made in the discussion that an annealed steel had shown as good results as a normalised one, and he thought that was probably because it was at a high temperature. He had noticed the same sort of effect with other materials and it seemed that the more active a material was made by working or heat-treatment, the more effective it was at lower temperatures and the less effective at higher. Exploratory heating tests were important in order to determine the behaviour of the material on exposure to high temperatures for a long time, but the essential test was one in which a series of creep tests were made at a certain stress in order to get some idea of the temperature at which the stress could be used in practice. The author had shown that chromium, up to 1%, had a beneficial influence in molybdenum steels and it might be thought that by lowering the carbon content they could take full advantage of the effect, but cracking experiences with low carbon molybdenum steels had made users chary of carbon contents below 0.10%. Before the war materials were in advance of steam turbine requirements, but higher temperatures were now being used and one case in U.S.A., with a temperature of 1,050°F., was near the limit for molybdenum-vanadium steel. Except as a means of ensuring adequate ductility he did not consider that rupture tests were of much value.

DR. H. A. DICKIE (Stewarts and Lloyds, Corby) referred to recent failures of molybdenum steel pipes in British power stations. Whilst local stressing, hammer marks and corrosive products from the lagging had all been given as unfavourable factors, he suggested that if the steel had had appreciable ductility it would not have failed. He welcomed the demonstration that 1% chromium greatly improved the ductility of molybdenum steel but felt it would be of value

if the minimum chromium necessary could be determined as the presence of 0.8% necessitated cooling at a slower rate than normal.

### Reply to Discussion

MR. J. GLEN said that in the time available he would only be able to reply to one or two of the points raised. He did not think that the effects of phosphorus on the creep rate would be very marked if the results were corrected for the effect of manganese. With regard to furnace-cooling, he considered that creep properties could be considered as due to the steel not being in equilibrium and that to approach equilibrium would destroy the creep properties. A furnace-cooled molybdenum-vanadium steel was worse in some respects than a molybdenum steel. In reply to Mr. Bardgett he thought his temperature of 550° C. was not much above the working temperatures now proposed for some of these steels. The paper was not intended to deal essentially with the properties of the steel at low temperatures, but to determine which steel would withstand the highest temperature without failure so as to give a factor of safety should the so-called service temperature be exceeded for a time. With regard to abnormal creep, he did not think there was a line of demarcation between normal and abnormal, the only way of checking it was by testing the same analysis steel in the two conditions. In reply to Dr. Bailey's remarks on rupture tests he would say that in dealing with a complex steel like molybdenum-vanadium steel, where it was difficult to recapitulate results, even if all were good, it was even more difficult to extrapolate to 100,000 hours and a rupture test could be used for confirmatory checking.

### A MICRO-EXAMINATION OF EIGHT STEELS FOR THE INCLUSIONS SUB-COMMITTEE.

By J. H. WHITELEY, D.Sc., F.R.I.C.

THE eight steels are all of the same type, nickel-chrome-molybdenum, made by the acid open-hearth, basic open-hearth and basic electric processes with varying degrees of cleanliness. The examination is part of an investigation into the effect of inclusions on fatigue strength, and Dr. Whiteley's findings will be more profitably studied in relation to the fatigue testing results when these are available. During the course of the investigation it was found that an inclusion count on a cross section gave results higher than would have been expected from the count on the longitudinal section, allowing for the increase due to the elongated nature of the inclusions. It is concluded that polishing removes some of the inclusions which are only embedded to a small depth, and an average of the two counts is taken as a measure of the cleanliness. Using this method, a linear relationship was found between the inclusion count and the percentage of sulphur and oxygen. The types of inclusion found in each steel are described and an appendix gives the results of an X-ray analysis by Mr. Murfitt on acid-extracted inclusions.

### THE STUDY OF SEGREGATIONS AND INCLUSIONS IN STEEL BY MICRO-RADIOGRAPHY.

By W. BETTERIDGE, B.Sc., Ph.D., F.Inst.P.,  
AND R. S. SHARP, B.Sc., L.I.M.

INCLUSIONS and segregation of alloying elements can be studied by radiographing slices of steel of 0.001-0.002 in. thickness, using a fine-grain film and subsequent enlargement. Details of the method of

preparation of the slices and of the radiographic technique are given. It is necessary to use a radiation such that the absorption coefficient of the element or compound is as different as possible from that of iron. For the elements with atomic numbers close to iron, the wavelength must be between the absorption edge of the element and that of iron. Where two elements segregate together, or form an inter-metallic compound, the effects are additive. Generally, with non-metallic inclusions the effect of the lighter element is such as to make the absorption in the inclusion less than in the steel and there is no possibility of identification by a choice of suitable radiations, although manganese containing inclusions are an exception. An extremely fine-grain film is necessary to permit enlargement up to  $200\times$  or even  $500\times$  without the grain effect becoming disturbing. Micro-radiography failed to reveal any grain boundary segregations in overheated steels, although some segregation was disclosed. It would seem that these are manganese rich areas and although they are not manganese sulphide, their sharpness suggests that they are not merely regions of manganese-rich solid solution. It is desirable that their nature and influence on mechanical properties be determined. In the case of ball race steels, segregation of manganese, chromium and possibly tungsten was disclosed, but no correlation between structure and running characteristics has yet been possible. The method is also of use in studying the segregation of elements in cast alloy steels.

#### SOME THERMODYNAMICAL ASPECTS OF THE FORMATION OF INCLUSIONS IN MILD STEEL WELD METAL.

BY E. C. ROLLASON, Ph.D., M.Sc. AND  
E. BISHOP, B.Sc.(TECH.)

THE authors have developed a method of thermodynamic analysis and applied it to published analyses of non-metallic inclusions in mild-steel arc-weld deposits. In spite of the short times involved in arc welding, equilibrium of the molten metal is largely attained at an estimated temperature of about  $1,710^{\circ}\text{C}$ ., and the process of inclusion formation in weld metal has been shown to obey the general principles of thermodynamics. Inclusions analysed at room temperature, and also calculated to exist at the freezing point are plotted on a ternary ( $\text{SiO}_2\text{-FeO-MnO}$ ) diagram, from which can be deduced the change in inclusion composition during cooling from the freezing point. Evidence is given to indicate that the oxygen content of the weld metal is determined by the FeO content of the slag, as in large scale steelmaking. Consequently a method has been evolved whereby the types and amounts of inclusions to be found in many weld metals can be determined by using the FeO content of the slag and the total manganese and silicon contents of the weld metal. Slags containing large quantities of calcium fluoride are excepted.

#### Joint Discussion

MR. H. H. BURTON (English Steel Corporation, Ltd.) said it was interesting to note that the micro-radiographic technique showed that there is sometimes more associated with inclusions than normal micrography suggests, and it might be instructive to apply the technique to the fatigue test specimens in the Inclusion Sub-Committee's work. He asked whether the weld metal samples in the last paper

were obtained by striking two rods together or from a weld run. In the latter case would not the parent metal influence the results?

DR. F. W. JONES (Brown-Firth Research Laboratories) remarked that to the X-ray crystallographer the use of these low wavelengths for exciting X-rays would appear at first ridiculous, but there was no doubt that such low voltages did give maximum contrast. Dr. Jones showed slides of micro-radiographs of manganese sulphide inclusions, grain boundary columbium segregation in a heat resisting steel, sigma phase, chromium-rich  $\delta$ -ferrite in 18% Cr. 10% Ni steel and chromium-rich  $\delta$ -ferrite in a duplex chromium-nickel-molybdenum corrosion resisting steel. The grain size of the steel seems to be a limiting factor, as even with finer grain films it would be necessary to have thinner specimens to deal with a grain size of less than 0.001 inch.

MR. K. J. B. WOLFE (B.S.A. Tool Group of Companies) said that they had used micro-radiography in a study of machinability. The machinability of a sample of oil hardened and tempered B.S. En. 23 steel was improved by a further tempering for an hour at  $25^{\circ}\text{C}$ . below the initial tempering temperature. Ordinary methods of investigation showed no apparent difference due to this re-tempering but micro-radiography showed severe manganese segregation which was less sharply defined after double tempering. It was considered that the original segregation was sufficiently marked to cause hard spots but with partial diffusion softening occurred. There was still sufficient difference from the matrix to allow the steel to machine more easily due to the manganese-rich regions acting as non-metallic inclusions do in free-cutting steel.

MR. H. A. SLOMAN (National Physical Laboratory) thought that the thermodynamic data available was not such that an accurate picture of the equilibrium in welding could be drawn, and that Messrs. Rollason and Bishop had over-simplified the problem as they had omitted to consider the effect of hydrogen and carbon. In the paper by Rooney, Schofield and himself, vacuum fusion, vacuum heating, alcoholic iodine and X-ray diffraction analyses had been used to obtain a clear picture of the problem, and he could not agree that the thermodynamic method gave a sufficiently accurate story. He gave a number of instances where the results of his work showed the inclusions to be different from those which would be predicted by the authors' method. It would be interesting to know whether the authors had any figures to justify the assumption that the total oxygen can be calculated from the FeO content of the slag. The paper was extremely interesting from an academic point of view, but he could not see its practical use, except to show the tendencies of the way in which inclusions might be formed.

DR. C. H. DESCH (London) referred to a micro-chemical technique developed at Cornell University by which inclusions could be identified by the formation of characteristic crystals when spots of certain reagents were applied to the inclusion under the microscope.

DR. L. MULLINS (Kodak Research Laboratories) emphasised that in micro-radiography the tone rendering may be very different from that of a photomicrograph, and that the method is restricted to the revelation of constituents which differ in X-ray absorption. As the plates are coated very thinly with an extremely fine-grain emulsion it is necessary to control the development very carefully, otherwise graininess may be developed. He queried the reference to the risk of halation near the edge of the specimen unless mounted on a piece of film, and asked whether the film merely served to reduce the tendency to irradiation round



the edge of the specimen and what increase of exposure was necessary to allow for the increased absorption of the composite specimen. Dr. Mullins suggested the investigation of two other techniques. In the first, 200 kv. radiation filtered by about 5 mm. of copper, strikes the specimen through the emulsion and different elements in the surface and immediate sub-surface give rise to different electron emissions. The second technique was the use of radioactive tracer elements for alloying, in which the specimen is left in contact with an extremely fine-grain film for some time, when a record is obtained of the distribution of the radioactive elements.

Owing to lack of time the President asked the authors to reply in writing.

#### A COMPARISON OF MOULDS OF STANDARD COMPOSITION AND OF APPROXIMATELY INGOT MOULD SUB-COMMITTEE COMPOSITION.

By W. L. KERLIE, B.Sc.

FOR the purpose of this investigation, 32 moulds of standard composition (P-0.1%) were compared with 32 moulds of approximately Ingot Mould Sub-Committee (I.M.S.C.) composition (P-0.2%). The silicon content of the latter was 1.4% instead of the specified 1.0-1.2%. Five moulds of each type were used on the same casts until they were scrapped when another five of each were tried. On this basis the I.M.S.C. composition was found to be slightly better. From the examination of the results by statistical methods it is concluded that, for I.M.S.C. composition, mould life increases with decreasing silicon content, but on the standard composition no such relationship has been established. Further data will be examined in an attempt to check this point. With a silicon content of 1.0-1.2 (I.M.S.C.) an average life of 130 may be expected as against 100 for the standard composition. The difference in phosphorus may account for the small improvement found, the data showing that increasing the phosphorus to at least 0.15% increases the expected life.

#### MOULD WEIGHT/INGOT WEIGHT RATIO AND ITS RELATION TO MOULD CONSUMPTION.

By N. H. BACON, A.MET., F.I.M.

STATISTICAL methods have been applied to the examination of very comprehensive data provided by three Ingot Mould Sub-Committee questionnaires (one pre-war and two post-war) and B.I.O.S. Report, No. 685, on German Ingot Moulds for the Casting of Steel Ingots. Very large variations in the M/I ratio were found, viz., 0.78-1.76 in Britain and 0.61-1.82 in Germany. It appears that there is an optimum M/I ratio for minimum mould consumption which varies with the size of mould. Up to 6 tons an approximate curve is given for the optimum value with 0.9 for 6 ton ingots and larger, 1.0 for 3 ton ingots, 1.2 for 2 ton ingots and 1.5 for 1 ton ingots. These figures do not include the steel in the feeder head and it is obviously necessary to use the curve as a general guide only, as there would be a difference with open- and closed-base moulds, for instance. The reasons for many moulds having a higher M/I ratio are probably the desire to increase the rate of solidification by a thicker mould (although this is open to question), the necessity to withstand rough handling in stripping and the desire to overcome major cracking failures, although such failures are more properly attributable to weak material and/or poor design.

#### FORMATION OF "DOUBLE SKIN" OR "CURTAINING" ON TOP POURED MILD STEEL INGOTS.

By P. WALKER.

THE paper describes work carried out to determine the cause of the ingot surface defect known as "double skin" or "curtaining." Two factors appear to be of significance, (a) the trapping of deoxidation slag near the ingot surface during solidification and (b) the formation of a "box" of splash which contracts from the mould wall and allows liquid steel to flow behind it. The former results in a "double skin" effect not visible on the surface and the latter a "double skin" or "curtaining" defect which is visible. During the experiments it was found that "curtaining" did not occur with tarred moulds nor with badly crazed moulds. The tar probably prevented the splash adhering, whilst with the crazed mould the splash is keyed to the wall and the liquid cannot get behind it.

#### Joint Discussion

DR. L. REEVE (Appleby-Frodingham Steel Co.) welcomed the growing tendency to use statistical methods in the examination of works data. The so-called I.M.S.C. composition was worked out for 4-ton ingots and his experience had shown that for the larger sizes a slightly higher silicon content is better. He questioned Mr. Bacon's rather arbitrary separation of his data at 130 cwt. ingot size and suggested that 180 cwt. would be more correct although it might not result in such good correlation. Those of 300 cwt. and upwards were obviously in a separate class. The formation of a "box" of splash on suddenly opening up the ladle nozzle, as described by Mr. Walker could be overcome by attention to teeming practice and in the case of larger ingots, by the use of splash cans.

MR. W. H. GLAISHER (B.I.S.R.A.) said he had done some further analysis on the data provided by Mr. Kerlie, and the result suggested that with silicon above 1.5%, phosphorus had little effect, but with silicon below 1.5%, high phosphorus (> 0.1%) gave better life. With regard to the effect of thicker walls on the rate of solidification, Nelson (A.S.M., 1934) had suggested that a 50% increase in wall thickness did not produce an appreciable rise in solidification rate.

MR. A. BEVAN (Guest Keen Baldwins Iron and Steel Co., Ltd.) said that they had found an improvement with low silicon, high phosphorus iron and had confirmed that the effect of phosphorus is greatest with low silicon values.

MR. KERLIE (Stewarts and Lloyds, Ltd., Corby), suggested that the M/I ratio should be based on that part of the mould which contained solid steel throughout so as to cater for solid-bottom and hot-top moulds. If Mr. Bacon's curve for optimum M/I ratio v. ingot mould size were made more or less asymptotic with regard to the mould axis, a curve would result whose extension would apply to small moulds.

#### Reply to Discussion

MR. N. H. BACON said he would justify in writing his action in considering only moulds up to 130 cwt. in size. He agreed with Mr. Kerlie that only that part of the mould containing solid steel should be considered in M/I ratios.

MR. P. WALKER said that he had not followed through to the finished product an ingot with "curtaining," but he proposed to do so. Mr. T. J. Wood, of the Republican Steel Corporation, Cleveland, Ohio, had written to say that he overcame the defect by what, in this country, would be regarded as very fast teeming indeed.

# Combating Corrosion of Iron and Steel

## The Scope and Practical Character of the Work of the Corrosion Committee of The British Iron and Steel Research Association and its Sub-Committees

**U**NDER normal peace-time conditions the need to conserve metals is very real, but, under the present abnormal conditions, it is an economic necessity to make the most prudent use of metals without waste or needless destruction. Conservation is not only concerned with the recovery of scrap metals and their use with or instead of metals produced from ores, but also with the preservation of structures with a view to extending their useful life. The main factor in promoting waste is corrosion and while all base metals suffer from this cause to a varying degree, it is particularly severe on iron and steel structures. The effect of corrosion on iron and steel in this country is estimated to involve a waste of more than a million tons annually. Obviously, the complete protection of iron and steel against corrosion would greatly increase the serviceable life of structures and enable our present production rate to meet more nearly the heavy demands for iron and steel, quite apart from the substantial labour economy effected by extending the time between renewals.

The problems of corrosion have been the subject of study in all industrialised countries for many years and increasing attention has, and is being given, not only to the application of remedies, but to the causes of the evil. The work of the Corrosion Committee, Sub-Committees and Joint Panels of the British Iron and Steel Research Association was discussed at a recent Conference at Birmingham, with Professor G. Wesley Austin, O.B.E., Chairman of the Corrosion Committee in the Chair. Supporting him were Mr. T. M. Herbert, Deputy Chairman Corrosion Committee, Chairman of Protective Coatings Sub-Committee; Mr. F. Fawcett, Chairman of Joint Technical Panels on Structural Steelwork, Anti-Fouling Compositions and Anti-Corrosion Compositions; Professor J. E. Harris, Chairman Marine Corrosion Sub-Committee; Dr. J. C. Hudson, Head of British Iron and Steel Research Association's Corrosion Laboratories at Birmingham; Dr. J. H. Jenkin, Chairman Industrial Waters Corrosion Sub-Committee; Mr. L. Kenworthy, Chairman Joint Industrial Panel on the Leaching Rate; Mr. T. H. Turner, Chairman Atmospheric Corrosion Sub-Committee; together with Mr. G. P. Acock, Mr. M. E. Davies, Cmdr. R. H. S. Rodger, R.N. (Retd.), and Mr. G. W. Withers, members of the staff of British Iron and Steel Research Association.

### The Work of the Corrosion Committee

The approach to solving corrosion problems is by the accumulation of data obtained either from particular cases occurring under service conditions, or from systematic investigations and tests carried out on particular metals and alloys or on groups of alloys. With both these means of obtaining data much work has been carried out on iron and steel by the Corrosion Committee and the results of investigations have not only added to knowledge of the troubles and failures

resulting from reactions in which metals are transformed into their compounds under the term corrosion, but have yielded data which have proved of value in the maintenance of metallic structures. As pointed out by Professor Wesley Austin, since the institution of the Corrosion Committee in 1928 much valuable work has been done and several reports have been published. Originally organised by the Iron and Steel Institute with the Iron and Steel Federation, it is now functioning under the auspices of the British Iron and Steel Research Association and the Corrosion Laboratories at Birmingham are under Dr. J. C. Hudson, who has been in charge of the Corrosion Laboratory since its formation in 1929. In discussing the work of this Committee, therefore, Dr. Hudson gave a broad outline of the manner in which the subject is being approached.

In studying corrosion problems, he said there are two broad methods of advance: the pursuit of fundamental investigations designed to discover new ideas and to provide a scientific background for the comprehension of the phenomena; and the conduct of tests and practical trials with the object of improving existing materials and processes.

The strength of the Corrosion Committee's organisation lies in the fact that it is well qualified to prosecute both these methods, as it is so constituted as to include both academic scientists and men with practical industrial experience within its membership. We look to the former, such as Dr. U. R. Evans, Dr. W. H. J. Vernon, O.B.E., and their colleagues to provide the fundamental background to our knowledge of corrosion and for new and novel ideas. Three examples of this fundamental work include:—

(1) The development by Dr. Evans and his collaborators at Cambridge of the so-called cementiferous paints. These are bound with an aqueous medium and the process of film formation closely resembles the setting of certain types of cement. Their protective properties when applied to steel exposed to certain types of corrosive conditions are remarkably good.

(2) The design by the team of investigators at the Chemical Research Laboratory, Teddington, under the direction of Dr. Vernon of the rotor test for the corrosion of metals immersed in sea water, aqueous solutions or other liquids. The specimens are mounted on the periphery of a rapidly rotating disc and the test has been proved particularly useful in simulating the exposure conditions of painting schemes applied to ships' bottoms moving through the water.

(3) The introduction by Professor J. E. Harris of a leaching rate test for the efficiency of anti-fouling compositions. As is well known, under certain conditions the hulls of ships become covered with marine growth. This increases the resistance to motion and, therefore either results in loss of speed or in increased fuel consumption to maintain a given speed. The method of remedying this defect is to paint the bottom of the ship

with a poisonous anti-fouling composition which prevents the settlement of the marine organisms. This composition only remains effective so long as the poisons, usually oxides or salts of copper and mercury, are liberated from the paint film into the seawater at an adequate rate. In its simplest terms, the leaching rate test is an accurate laboratory method of studying how this rate of liberation varies with time and it affords a useful means of predicting the probable service behaviour of an anti-fouling composition.

The conduct of the practical exposure tests, service trials and the general work of perhaps slightly more immediate application to industrial needs falls to the B.I.S.R.A. Corrosion Laboratory. I should like to say at once that in this work we enjoy the benefits of unstinted support and collaboration from our own industry and from those industries and other research bodies who are collaborating with us, as well as from individual members on our research committees. The assistance chiefly takes the form of the supply of materials or services and has the effect of materially increasing the scope of the work that my laboratory would be able to undertake under present conditions with its own unaided efforts. As a simple illustration of this, the preparation and exposure of a recent series of test specimens involved a total man-power expenditure of about 90 man-days. Of this total, only about 20 man-days was supplied by the B.I.S.R.A. Corrosion Laboratory; the remainder was given by staff seconded for the purpose by the representatives of other organisations collaborating in the research.

Continuing, Dr. Hudson said, it is the ambition of the Committee to investigate and combat the rusting of iron and steel in all fields of service. To this end tests are being conducted on the comparative resistance of different types of iron and steel to corrosion and on the efficiencies of various protective schemes for ferrous metals in a number of different media, including the atmosphere, the sea, the soil and industrial waters.

The Atmospheric Corrosion Sub-Committee, under the chairmanship of Mr. T. Henry Turner, has shown that the life of ordinary structural steel exposed to the atmosphere without a protective coating may be increased as much as threefold by the addition to the steel of small quantities of alloying elements, such as copper and chromium, without any undue increase in cost, considered in relation to the improved performance of the material.

The Protective Coatings Sub-Committee, under the chairmanship of Mr. T. M. Herbert, has conducted several extensive researches, the results of which have materially added to our knowledge of many types of protective coatings for iron and steel, more especially of painting schemes and metallic coatings used for protection against atmospheric corrosion. More recently, the Sub-Committee has entered into intimate collaboration with the paint and bituminous protective products industries and with the assistance of the representative organisations of these industries has set up a joint technical panel under the chairmanship of Mr. F. Fancutt to make a detailed study of protective painting schemes for steelwork exposed to the atmosphere.

The Marine Corrosion Sub-Committee, with Professor J. E. Harris as chairman, has made a detailed study of the prevention of corrosion by sea water and the prevention of fouling. These researches are particularly applicable to the underwater protection of steel ships.

In the former case, we may justly claim to have made considerable advances in the development of efficient protective paints. As a result of a series of researches on behalf of the Sub-Committee, a number of anti-corrosive compositions of proved performance have been developed which are regarded as setting a reasonable standard of efficiency for protection in this field. One of these has been used by the Admiralty with satisfaction and, the outer hull plating of the two large ships, the *Pretoria Castle* and the *Edinburgh Castle*, constructed at Belfast by Messrs. Harland and Wolff Ltd. for the Union-Castle Mail Steamship Company Ltd. was painted on the stocks with compositions and paints to the Sub-Committee's formulations. The second ship has not yet been dry-docked after fitting out but the *Pretoria Castle* docked some weeks ago, about 7 months after launching. On inspection, 98% of the bottom paint was found to be perfectly intact and unstained by rust. This was a remarkable result and both the owners and the builders were favourably impressed by it. Incidentally, this is a good example of the manner in which, with the collaboration of the industries using steel, the Corrosion Committee is able to arrange for service tests on an adequate scale.

The Industrial Waters (Corrosion) Sub-Committee, under the chairmanship of Dr. J. W. Jenkin, is responsible for the study of corrosion in the field indicated by its title, which includes all types of water installations. It has compiled a short popular brochure surveying knowledge and practical experience of corrosion by industrial waters, which should be published towards the end of this year.

Extensive tests on the corrosion of iron and steel in the soil were begun some years ago by the Sub-Committee on the Corrosion of Buried Metals, in collaboration with the British Non-Ferrous Metals Research Association and the Institution of Civil Engineers. The first sets of specimens are due for removal from the sites at which they have been buried in the summer of 1949.

### Practical Applications of the Work

It is appropriate to add a few details to indicate the manner in which the results of these investigations receive practical application in industry. In the course of a year, said Dr. Hudson, we receive, either directly or through our User Liaison Department at Headquarters, over fifty requests for advice regarding corrosion problems, the majority of which relate to industrial equipment or plant. He illustrated these by giving the bare headings of some of the most interesting cases with which we have dealt during the past year.

The range of subjects varies from the protective schemes to be used for the steelwork of the new Severn Bridge, or for painting electricity standards in Siberia, to the problem of reducing the corrosive effects of hydrofluoric acid in the flue gases of pottery kilns. Advice has been sought regarding the rapid corrosion of the steel cars used for quenching coke, the protection of a steel pipe line to be used for a hydro-electric scheme in Scotland and the prevention of corrosion in steel barges. An unusual enquiry, to which unfortunately no practical solution could be suggested, was received from the War Office who asked for advice as to how to speed up corrosion. They were concerned with the disposal of the masses of barbed wire entanglements erected during the war, which it is impracticable to remove and must be left to rust to destruction.



Two Corporations sought advice regarding the corrosion or protection of their municipal piers and a third requested views on the durability of a cast iron or steel water main to be laid in the silt at the bottom of a dock. The National Coal Board sent in an enquiry regarding the use of low-alloy steels for pit tubs. We have been asked to advise on the protection of steel kitchen/bathroom units, now widely used in prefabricated housing construction, of galvanised steel sinks and of overhead line switchgear for use in the tropics. In the field of marine corrosion, a general request was received for information on marine paints from the High Commissioner of one of the Dominions and a more specific enquiry from one of our main shipping companies with reference to the painting of a new liner.

Other interesting examples were given by Dr. Hudson to show the great practical importance of this work on corrosion. Despite the growing attention that is paid to the results of this work, it is felt it is still not sufficiently well known throughout industry generally. In the national interest steel should be used as economically as possible, particularly under present circumstances where the demand for steel far exceeds the possibilities of production.

### Protective Coatings

The fight against corrosion is as vital as the campaign for scrap recovery, said Mr. T. M. Herbert, and the prolongation of the life of structures aids the national economy in avoiding the premature use of manpower and material required for renewals. It may seem strange that a Corrosion Committee set up and supported by the steel industry should undertake research on protective methods, but the metallurgist still seems a long way from producing a cheap non-corrodible structural steel, and it was therefore a realistic action to appoint a Sub-Committee in 1937 to study methods of protecting ordinary mild steel. This initial step of bringing together the three parties concerned—the steelmaker, the manufacturer of the protective coating, and the engineer as ultimate user—to form with those engaged in the scientific study of the problem, a disinterested and impartial body to study the protection of steel by all or any of the various methods available, has proved very successful.

The work so far performed or in hand has covered three main subjects:—

(1) Methods of pre-treating iron and steel surfaces prior to painting. The results of work done show the importance of spending a certain amount of effort and money in removing rust and millscale, preferably by pickling or by sand- or shot-blasting, and where appropriate by flame cleaning. Unfortunately, the advice in some respects is so simple as to be ignored in practice.

(2) Painting schemes suitable for brush application to steel work prepared under average industrial conditions. For these tests a large number of formulated paints will be specially prepared. The initial programme covers these main lines: A range of primers, followed by a standard finishing coat; a range of finishing coats over a standard primer; and bituminous and allied coatings.

(3) Metallic and other non-paint protectives. Here the object was to compare impartially, under precisely similar conditions, the behaviour of various forms of metallic coatings—dipped, sprayed or electro-deposited as the case may be—both with each other and with the

more traditional used paint protection. Certain less usual protectives, including vitreous enamel are included.

No other body can bring together, in an impartial review, all the known methods of protecting steel against corrosion, and it is in this unique catholicity of outlook, and in the voluntary assistance given by members and their organisations, that its value largely resides. The appended bibliography of recent published work will assist those seeking detailed information of investigations carried out.

### BIBLIOGRAPHY.

- "Protective Painting of Structural Steelwork," submitted by the Protective Coatings Sub-Committee. *J.I.S.I.* 1942, No. 1, p. 87P.
- "Progress in the Corrosion Research Section at Cambridge University," by U. R. Evans. *J.I.S.I.* 1944, No. 1, p. 67P.
- "Fouling of Ships' Bottoms: Identification of Marine Growths," submitted by Marine Corrosion Sub-Committee. *J.I.S.I.* 1944, No. 11, p. 143P.
- "The Formulation of Anti-Corrosive Compositions for Ships' Bottoms and Under Water Service on Steel—Part I," by F. Fancutt and J. C. Hudson, submitted by the Marine Corrosion Sub-Committee. *J.I.S.I.* 1944, No. 11, p. 269P.
- "The Formulation of Anti-Corrosive Compositions for Ships' Bottoms and Under Water Service on Steel—Part II," by F. Fancutt and J. C. Hudson. *J.I.S.I.* 1946, No. 11.
- "Interim Descriptive Statement on the Leaching Rate Test for Ships' Anti-Fouling Compositions," submitted by the Joint Technical Panel on the Leaching Rate Test. *J.I.S.I.* 1946, No. 11.
- "Report on Anti-Fouling Research 1942-44," submitted by the Marine Corrosion Committee. *J.I.S.I.* 1946, No. 11.
- "The Protection of Iron and Steel by Metallic Coatings," by J. C. Hudson and T. A. Banfield. *J.I.S.I.* 1946, No. 11.
- "Interim Report on the First Series of Tests on Formulated Priming Paints for Structural Steelwork (R328)," Joint Technical Panel J/Pl.—Paints for Structural Steelwork.
- "The Corrosion of Iron and Steel and its Prevention," by J. C. Hudson. *J.O.C.C.A.*, XXX, No. 329, February, 1947.
- "The Protection of Ships' Bottoms and the Formulation of Anti-Corrosive Compositions," by F. Fancutt and J. C. Hudson. *J.O.C.C.A.*, XXX, No. 323, May, 1947.
- "Chemical Research and Corrosion Control: Some Recent Contributions of a Corrosion Research Group," by W. H. J. Vernon. *J.S.C.I.*, May, 1947.
- "Influence of Residual Stress on Chemical Behaviour," by U. R. Evans. Institute of Metals Monograph and Report Series No. 5.
- "La Protection des Coques de Navires Contre la Corrosion et la Mise au Point de la Composition des peintures anti-corrosion," by F. Fancutt and J. C. Hudson. Peintures, Pigment, Vernis, October and November, 1947.
- "Service Tests of Experimental Anti-Fouling Compositions," by H. Barnes, M. W. H. Bishop and E. A. Pyefinch. *J.I.S.I.*, November, 1947.
- "Performance Test for Protective Schemes Embracing Stoving Paints Used in the Protection of Light-Gauge Steel and Wrought Iron against Corrosion," B.S. Institution. (P.B.S. 1391:1947.)
- "The Prevention of Corrosion on Structural Steelwork," by F. Fancutt and J. C. Hudson. Institution of Civil Engineers, February, 1947.
- "Corrosion—A Summary of the Work of the Joint Committees," by J. C. Hudson. *Iron and Steel*, November, 1947.
- "The Protection of Steel by Various Non-Metallic Coatings," by J. C. Hudson and T. A. Banfield. *J.I.S.I.*, January, 1948.
- "Cementiferous Paints," by J. E. O. Mayne and R. S. Thornhill. *J.I.S.I.*, February, 1948.
- "Marine Exposures of Cementiferous Painting Schemes," by K. A. Pyefinch. *J.I.S.I.*, February, 1948.
- "The Measurement of Paint Film Thickness on Mild Steel," by F. Fancutt and J. C. Hudson. *Paint Technology*, 1948, XIII, No. 146.
- "First Report of the Methods of Testing (Corrosion) Sub-Committee." *J.I.S.I.*, April, 1948.

## Liverpool Metallurgical Society

As a result of an initial meeting in Liverpool approximately 120 people expressed their intention of joining the proposed Liverpool Metallurgical Society. A Committee has been formed and it has been decided not to hold another meeting before the autumn, but to concentrate on the preparation of a syllabus for the 1948-49 session. The Committee propose to arrange six lectures at monthly intervals commencing in October, to include joint meetings with the Liverpool Engineering Society and the N.W. Section of the Society of Chemical Industry. An annual general meeting is proposed for April, 1949. All interested, who have not already made contact, should communicate with Mr. C. W. J. Gellatley, 7, Woolacombe Road, Liverpool, 16, and assist in making this a really live organisation.

## Institution of Metallurgists

THE Third Annual General Meeting of the Institution of Metallurgists was held at the offices of the Institution, 4, Grosvenor Gardens, London, S.W.1, on Thursday, 10th June, at 3.0 p.m. Following the adoption of the Report of Council and the Treasurer's Statement, the Retiring President, Dr. J. W. Jenkin, announced that the following officers had been elected:—

*President*—Maurice Cook, D.Sc., Ph.D.

*Vice-Presidents*—E. W. Colbeck, M.A. and A. J. Murphy, M.Sc.

*Hon. Treasurer*—C. J. Smithills, M.C., D.Sc.

*Members of Council Representing the Fellows*—

N. P. Allen, M.Met., D.Sc. and L. B. Pfeil, O.B.E., D.Sc., A.R.S.M.

*Member of Council Representing the Associates*—

L. Rotherham, M.Sc., F.Inst.P.

Dr. Jenkin said that he was laying down the reins of office with a certain amount of satisfaction, in that the Institution was making substantial progress and in that he was handing over to a successor who commanded the complete confidence of the members. The membership of the Institution was now about 1,700 and recognition was continually growing. The Institution's qualifications had been specified by Government Departments and industrial organisations in announcing metallurgical vacancies. In the field of education, the Institution was represented on the Joint Committee on National Certificates in Metallurgy, the Joint Committee on Metallurgical Education and the Mond Nickel Fellowship Committee, as it was also on the Parliamentary and Scientific Committee and the Joint Council of Professional Scientists.

Dr. Jenkin referred to the appointment of Dr. A. D. Merriman as Registrar-Secretary and paid tribute to the work of Mr. Headlam-Morley and Dr. Moore to whom the Institution owed a great debt for their services in the very difficult early days. He was pleased to announce that the Council had elected Dr. Moore as its first Honorary Member.

In handing over to the new President, Dr. Jenkin said that Dr. Cook was a metallurgist of the highest standing, holding a very responsible post with the Metals Division of I.C.I., Ltd. Apart from his technical work, he had a very wide administrative experience on various metallurgical Councils. He brought to the office a combination of wisdom, tact and courage that would make his Presidential year memorable.

Dr. Cook expressed his appreciation of the confidence of the members in inviting him to serve as President and said it was surprising that metallurgists, concerned with a branch of human activity going back beyond the dawn of history had only just formed their own professional organisation, but he thought the success already achieved belied the doubts and fears of those who had thought the Institution to be unnecessary. What they lacked in tradition was made up for by zeal and energy and they must, as the years go by, establish a tradition worthy of metallurgists of our generation.

It was hoped that those seeking membership would not do so from any selfish motives, but rather would do so in order to lend their aid and support in achieving the aims and objects of the Institution, so that metallurgists in the years to come would join in a spirit of service and collective endeavour to advance the interests and well-being of the profession.

It should be the aim of the Institution to have as members, all the metallurgists in the country with the necessary qualifications, but in the interests of the profession, a high standard of competence should be required. He had stressed the importance of motives and ideals, for if they had any awareness at all of realities, they must appreciate that the major problems of the day were not material, but idealistic and of the spirit. Only if their ideals were of the highest would they merit and achieve success.

## Institute of British Foundrymen

THE Forty-fifth Annual Conference of the above Institute was held at the Café Royal, Regent Street, London, W. 1, on June 9-11 inclusive. The Conference opened on June 9 with the Annual General Meeting, the President, Mr. P. H. Wilson, O.B.E., being in the Chair, at which Reports of the Council and of the Technical Committee were presented, together with a statement of the financial position of the Institute.

Mr. R. B. Templeton, M.I.Mech.E., was elected as President and Messrs. N. P. Newman, J.P. and J. J. Sheehan, B.Sc., as Senior and Junior Vice-Presidents respectively for the year 1948-49. During the course of the meeting the following medals were presented: The Oliver Stubbs Gold Medal to Mr. L. W. Bolton, the E. J. Fox Gold Medal to Mr. J. G. Pearce and the Meritorious Services Medal to Mr. C. Lashly, M.C.

Mr. Templeton was inducted into the Chair and delivered his Presidential Address. The morning session concluded with the Edward Williams Lecture, delivered by Dr. H. Schwartz, of Cleveland, Ohio, on "Some Solved and Unsolved Problems in the Metallurgy of Blackheart Malleable."

The following eight papers were presented for discussion in the afternoon of June 9th: "Nodular Cast Irons, Their Production and Properties," by H. Morrogh and J. W. Grant; "A Quantitative Study of the Residual Gases in Cast Iron," by J. E. Hurst and R. V. Riley; "Plastic Flow in Cast Iron at Room and Elevated Temperatures, with Special Reference to Relief of Stress," by C. R. Tottle; "The Maurer Diagram and Its Modifications," by H. Laplanche; "Steel Castings for Aircraft," by E. J. Brown and F. Rogers; "Steel Casting Cleaning Methods," by A. B. Lloyd; "Gas Porosity in Nickel-Silver Castings," by T. F. Pearson, W. A. Baker and F. C. Child; "Aluminium Alloy Casting Developments," by E. G. West; whilst the morning of June 10 was occupied by the discussion of a further eight papers, viz.: "Refractory Materials in the Foundry Industry," by A. T. Green and G. R. Rigby; "Problems of Contraction and Distortion in Iron Castings," by E. Longden; "New Ladle Methods of De-sulphurising Pig Iron," by W. C. Newell, A. G. Langner and J. W. Parsons; "Bath Making," by A. Young; "Some Fundamental Problems in Foundry Planning," by Jorgen Drachmann; "Do You Use Your Cost System?" by Ralph L. Lee; "Metallurgy in Relation to Foundry Practice," by J. Barrington Stiles; and "Application of the Brinell Test in the Foundry," by J. Leonard.

THE full title and location of McKechnie Brothers' South African Company is as follows: McKechnie Brothers (South Africa) (Pty.), Ltd., 42/43, Rendell Road, Wadeville Industrial Sites, Germiston, Transvaal.

# The Chemical Analysis of a Permanent Magnet Alloy

By C. H. R. Gentry

Material Research Laboratory, Philips Electrical Ltd.

*The manufacture of permanent magnet alloys possessing specified magnetic properties requires rigid control of the composition and implies rapid and precise analytical checking of the melts. The determination of several constituents presents difficulties; some of these difficulties are described and procedures are given for the determination of cobalt, nickel, aluminium, copper, titanium, silicon and manganese. A significant feature is the use of photometric and polarographic methods for the determination of elements present in high percentages.*

PERMANENT magnet alloys containing 15-30% cobalt, 12-20% nickel, 5-10% aluminium, 2-7% copper, 0-10% titanium, 0-1% silicon, 0-0.2% manganese and the remainder iron,\* are a class of material of increasing industrial importance. The manufacture of such alloys with specified magnetic properties requires rigid control of the composition, and implies rapid and precise analytical checking of the melts. However, the determination of the several constituents of these alloys by the usual procedures employed in steel analysis presents a number of difficulties, and the establishment of simpler and speedier methods of control is desirable.

Over a period of several years a number of procedures have been attempted for determining the various constituents, and it would appear to be of some value to describe the difficulties which have been encountered as well as the methods of analysis which have been developed.

## Cobalt

The most generally recognised method for the determination of cobalt in steels involves a zinc oxide separation and final precipitation with a nitroso  $\beta$  naphthol.<sup>1,2,3</sup> The presence of alloying amounts of copper and nickel in the steel complicates the procedure; copper must be separated with hydrogen sulphide, and to effect separation of cobalt from nickel it is necessary to carry out a double precipitation with a nitroso  $\beta$  naphthol. It will be realised that the application of this method to the analysis of magnets is somewhat tedious and attention has, therefore, been given to more rapid procedures. Several photometric methods are available for determining small amounts of cobalt in steel, but the application of such methods to alloys containing 25% cobalt requires that the solution of the sample should be made up to a standard volume, a small aliquot taken and the dilution repeated until an aliquot is obtained which contains the necessary quantity of cobalt. The successive dilutions are tedious and may lead to a lack of precision in the final result. However, the photometric determination of cobalt with ferri-cyanide in ammoniacal solution<sup>4,5</sup> is not so sensitive as some of the other procedures, and a successful method

for the analysis of the magnet alloy has been based on it. The procedure finally adopted may be briefly described as follows:—

Dissolve 1 gm. of sample in 100 mls. of acid mixture (400 mls. hydrochloric acid and 100 mls. nitric acid diluted to 1 litre). Boil off nitrous fumes, and dilute to 250 mls. in a graduated flask. Allow any silaceous residue to settle. Pipette 10 mls. of this main solution into a 100 mls. graduated flask. Add in this order, mixing between additions, 10 mls. ammonium citrate solution (675 mls. conc. ammonium hydroxide added to 500 gms. citric acid dissolved in a litre of water), 20 mls. 1:1 ammonium hydroxide, 10 mls. 1% potassium ferri-cyanide and water to the mark. Set the Spekker absorptiometer water-to-water 1.00 with 1 cm. cell and 603 filters. Fill 1 cm. cell with solution and take drum reading. Determine percentage of cobalt from standard graph.

The standard graph, Fig. 1, is prepared from synthetic solutions containing accurately known amounts of spectrographically pure cobalt, and the quantities of iron, nickel and copper corresponding to typical magnet solutions.

The average deviation found between the results of photometric cobalt determinations and referee chemical

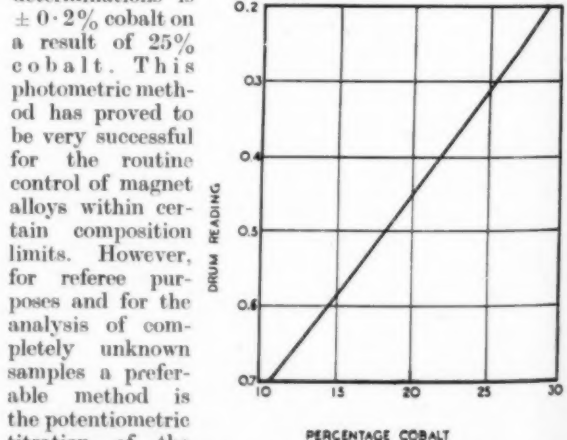


Fig. 1.—Calibration graph for cobalt. The cobalt with potas- sium ferri-cyanide in ammoniacal citrate solution.<sup>6</sup> This method, which has only recently received much attention in this country, is rapid, accurate and reliable.

\* The work reported in this paper was carried out with permanent magnet alloys known under the Registered Trade Mark "Ticonal."

1 (Chemists of U.S. Steel Corp.) "Sampling and Analysis of Carbon and Alloy Steels," Reinhold Pub., 1928.

2 "Standard Methods of Analysis," United Steel Co., Ltd., 3rd edition, 1945.

3 E. C. Pigott, "The Chemical Analysis of Ferrous Alloy," Chapman & Hall, 1942.

4 W. J. Angew. Analot. 1928, 53, 31.

5 R. J. De Gray and E. P. Rittershausen, Ind. Eng. Chem. (Anal. Ed.), 1942, 14, 858.

6 Steele and Phelan, Gen. Elect. Review, 1939, 42, 218.



The only interfering element present in the alloy is manganese, which titrates completely with the cobalt. The necessary correction is readily applied, although in most cases it is negligible.

The method used may be described as follows:—

Place 100 mls. of buffer solution (675 mls. conc. ammonium hydroxide added to 500 gms. of citric acid dissolved in a litre of water) and 80 mls. of conc. ammonium hydroxide in a 400 ml. tall-form beaker. Insert platinum (+ ve) and saturated calomel (— ve) electrodes, and a mechanical stirrer. Connect electrodes to terminals of a Mullard potentiometric titration apparatus E 920. Set the dial to read 30 millivolts. Add to contents of beaker from a pipette, 25 mls. potassium ferricyanide solution (15 gms. per litre). Add slowly 50 mls. of the main magnet solution, prepared as previously described. Back titrate with standard cobalt nitrate solution (10 gms. per litre) until the "magic eye" opens. Standardise the potassium ferricyanide solution by titrating 25 mls. with the cobalt nitrate solution using the above method, omitting the sample. Standardise the cobalt nitrate solution by direct analysis, or preferably by the above method using a suitable aliquot, containing 0.05 gms. cobalt, of a standard solution prepared from pure cobalt metal.

$$\% \text{ Cobalt in sample} = \frac{250}{50W} \times 100 (a-b) \times$$

where, 25 mls. potassium ferricyanide solution = a mls. cobalt nitrate solution.

1 ml. of cobalt nitrate solution contains x gms. cobalt.

Back titration with cobalt nitrate solution = b mls. and Original sample weight = W gms.

#### Nickel

The chemical determination of nickel in steels is either made volumetrically, by the silver nitrate—potassium cyanide method, or gravimetrically with dimethylglyoxime.<sup>1,2</sup> Whilst both these methods are subject to interference from copper and cobalt the latter method is more readily adapted to overcome these interferences. The co-precipitation of copper with nickel dimethylglyoxime can be reduced by using additional reagent, and effectively eliminated by carrying out a second precipitation. The interference of cobalt in the presence of ferric iron is more serious, and in the author's experience with magnet alloys a double precipitation will not give a pure nickel dimethylglyoxime precipitate. A more satisfactory method of eliminating cobalt interference is to precipitate in sodium acetate-acetic acid solution, keeping the iron reduced with sulphur dioxide.<sup>7</sup> A double precipitation of nickel dimethylglyoxime under these conditions gives a reasonable separation from cobalt and copper. A further method of reducing the cobalt interference is to oxidise it to the cobaltic state with hydrogen peroxide in ammoniacal tartrate solution.<sup>3</sup> By carrying out a double precipitation in this manner the nickel content of magnets can be satisfactorily determined.

A detailed description of this chemical method will not be given, as it has been found that the routine determination of nickel is best made either photometrically or polarographically.

The photometric method is essentially that of Vaughan,<sup>8</sup> modified to permit the determination of the

high percentages of nickel in magnets. The method may be briefly described as follows:—

Pipette 10 mls. of the main solution (as for cobalt) into a 100 ml. graduated flask and dilute to the mark with water. Pipette two 10 mls. portions of the diluted solution into 100 ml. graduated flasks A and B. Add to A, in this order, with mixing after each addition, 10 mls. ammonium citrate solution (as for cobalt), 30 mls. water, 5.0 mls. 0.1 normal iodine solution, 20.0 mls. ammoniacal dimethylglyoxime reagent (1 gm. of dimethylglyoxime dissolved in 500 mls. of conc. ammonium hydroxide and diluted to 1 litre with water), and water to the mark. Add to B, in this order, 10 mls. ammonium citrate reagent, 30 mls. water, 5 mls. 0.1 normal iodine, 20 mls. 1:1 ammonium hydroxide and water to the mark. Set Spekker absorptiometer water-to-water 1.00 with 602 filters and take drum readings of colour and blank solutions using 1 cm. cells. Determine percentage of nickel from difference.

The standard graph, Fig. 2, is prepared from synthetic solutions corresponding to typical magnet alloy compositions.

To obtain precise results by this method it is important that all the reagents should be measured out accurately, and well mixed before further additions are made. The dimethylglyoxime reagent deteriorates with keeping, and it is essential that the stock solution of this reagent should be prepared at frequent intervals. However, when these precautions are observed the method is rapid, well adapted to batch determinations and capable of giving an accuracy of  $\pm 1\%$  of the nickel content of the alloy.

The polarographic method for the determination of nickel is based on the work of Lingane and Kerlinger.<sup>9</sup> In the first place, attempts were made to determine copper, nickel and cobalt simultaneously by the polarographic procedure. For certain types of magnet alloy this very rapid method of analysis is possible. Unfortunately the results obtainable lack the required precision, particularly in the case of cobalt where the polarographic wave is not suitable for accurate measurement. Furthermore, the suppression of the very marked maxima

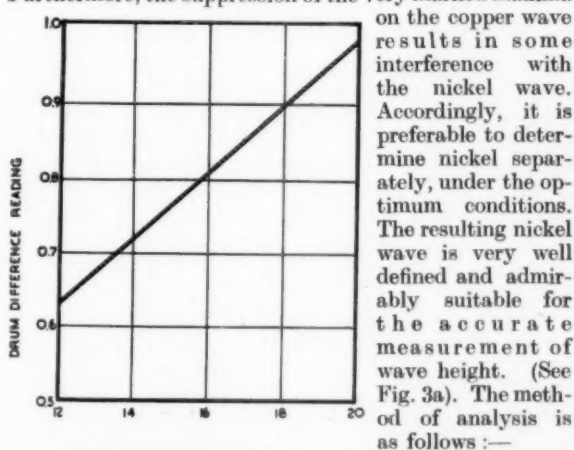


Fig. 2.—Calibration graph for nickel. Pipette 10 mls. of the main solution (as for cobalt) into a 100 ml. beaker, and evaporate to incipient dryness. Add 2.0 mls. 12N hydrochloric acid, and warm until the salts dissolve. Transfer to a 100 ml.

<sup>7</sup> J. G. Weidenburg, *Rec. trav. chim.* 1924, **43**, 465.

<sup>8</sup> S. J. Vaughan, "The Use of the Spekker Photo-Electric Absorptiometer in Metallurgical Analysis," Roy. Inst. of Chem. Monograph.

<sup>9</sup> J. J. Lingane and H. Kerlinger, *Ind. Eng. Chem. (Anal. Ed.)* 1941, **13**, 77.

graduated flask with 60 mls. of water. Add 5.0 mls. pyridine, mix and add 5 mls. 1% gelatine solution. Dilute to the mark. Allow the precipitate to settle somewhat and pipette a portion of the supernatant liquid into a polarographic cell. Place the cell in the thermostat and gas the solution with hydrogen for five minutes. Record polarogram on Cambridge Scientific Instrument Co., polarograph, using a voltage range of  $-0.6$  to  $-1.1$ , no counter current, damping 2, and galvanometer sensitivity  $\frac{1}{100}$ . Measure wave height and hence calculate the nickel percentage.

This method has been tested on a series of synthetic solutions corresponding to a wide range of magnet alloy compositions. It has been found that the ratio wave height at full galvanometer sensitivity divided by the percentage of nickel on the basis of a 1.000 gm. sample, is constant to within  $\pm 1\%$ . It has also been established that the average deviation between referee chemical determinations and polarographic determinations is  $\pm 0.1\%$  nickel on a result of 15% nickel.

Of the two methods for nickel which have been described in detail, neither is markedly superior in speed or precision to the other. For batch analysis the photometric method is preferred, as the instrumental measurements occupy less time; but for occasional determinations the polarographic method has the advantage that fewer reagent solutions are required.

#### Aluminium

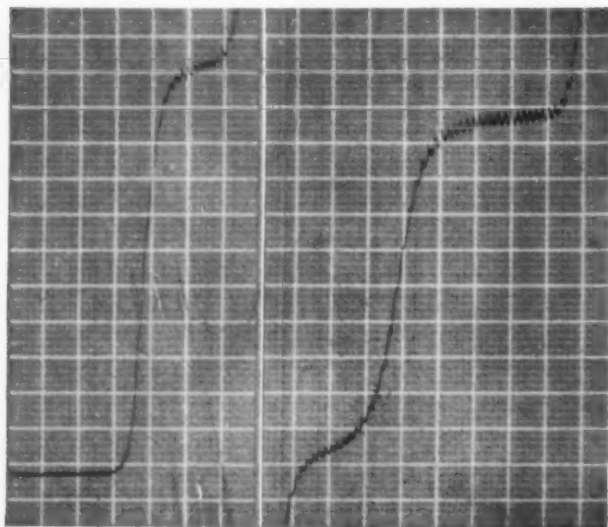
The rapid determination of aluminium in permanent magnet alloys containing titanium has proved to be a somewhat difficult problem. For occasional determinations, mercury cathode electrolysis has been employed to separate the other alloying constituents, and leave a solution in which aluminium and titanium are readily estimated. However, the use of mercury cathode electrolysis for batch analysis is not attractive, and attention has, therefore, been given to other procedures. Of the numerous methods which have been suggested for determining aluminium in complex steels, the simplest would appear to be that based on the precipitation of aluminium with 8-hydroxyquinoline

after the conversion of the heavy metals to complex cyanides in ammoniacal tartrate solution.<sup>10,11</sup> In the absence of titanium this method is satisfactory despite a small amount of co-precipitation which could not be neglected in very accurate work. However, the presence of titanium, which is also partially precipitated by 8-hydroxyquinoline under the conditions used, necessitates a modification of the method.

Pigott<sup>3</sup> proposes a method in which the precipitation of aluminium hydroxyquinolate is made from acetic acid solution after the cyanide treatment. It is claimed that by this means a single precipitation will give a separation of aluminium from titanium. In the author's experience, this method is not suitable for the analysis of the permanent magnet alloy, and it is necessary to overcome the titanium interference either by a preliminary separation or by suitable treatment of the mixed aluminium and titanium hydroxyquinolate precipitate.

The preliminary separation of titanium can be made by one of several methods—e.g., by the sulphur dioxide method of Baskerville, with cupferron, or by the sodium hydroxide method. The removal of titanium together with iron and copper by cupferron<sup>1</sup> followed by the determination of aluminium by the cyanide-oxine procedure is an accurate method, but it is rather time-consuming.

The best method of determining aluminium involves the precipitation of aluminium and titanium by the cyanide-oxine procedure, solution of the precipitate in dilute mineral acid, treatment with an excess of sodium hydroxide, and estimation of aluminium in the filtrate after removing the precipitated hydrous titanium dioxide. The method may be described thus: Pipette 50 ml. of the main magnet solution (as for cobalt) into a 400 ml. beaker. Add 2 gms. ammonium chloride and 4 gms. tartaric acid. Dilute to 100 mls. and add dilute ammonium hydroxide until the solution ceases to darken. Add 5 gms. potassium cyanide. Bring to the boil rapidly and boil for 1 minute. Cool to  $70^{\circ}\text{C}$ ., and add dropwise 12 mls. 5% alcoholic 8-hydroxyquinoline with good stirring. Add 5 drops of a 1% aqueous solution of sulphonated lorol. Digest at  $70^{\circ}\text{C}$ . for 10 minutes, cool to  $50^{\circ}\text{C}$ . and filter through a Whatman 541 paper. Wash several times with warm water and discard the filtrate and washings. Wash the precipitate back into the original beaker with a jet of water, and dissolve any precipitate adhering to the paper with 15 mls. of hot 3N hydrochloric acid. Wash the paper four times with water, and discard it. Heat the precipitate and combined washings, until all the precipitate dissolves. Add 20 mls. 25% sodium hydroxide solution and boil for 1 minute. Digest hot for 5 minutes and filter immediately through a Whatman 540 paper. Wash the precipitate with 2% sodium hydroxide, and discard it. Dilute the filtrate to 200 mls. Add 1 gm. tartaric acid, 1 gm. potassium cyanide, 3 mls. 5% alcoholic 8-hydroxyquinoline and 5 drops of sulphonated lorol. Heat to boiling, add with stirring, solid ammonium chloride until the oxine precipitation begins to form and then 5 gms. excess. Stir for 1 minute and digest at  $70^{\circ}\text{C}$ . for 10 minutes. Filter through a weighed sintered glass crucible. Wash with warm water. Dry at  $110^{\circ}\text{C}$ . for 1 hour, cool and weigh. The precipitate contains 5.87% aluminium.



(a) Polarogram of nickel; (b) polarogram of copper.  
Fig. 3.

<sup>10</sup> T. Hecako, *Chem. Ztg.* 1934, **58**, 1032.

<sup>11</sup> E. C. Pigott, *J. Soc. Chem. Ind.* 1939, **58**, 139.

This method may be simplified if the magnet alloy contains no titanium, by filtering the first aluminium hydroxyquinolate precipitate through a weighed sintered glass crucible, washing, drying and weighing. A slight modification of the method is necessary when the alloy contains a high percentage of titanium, greater than about 3%. In this case, the addition of extra 8-hydroxyquinoline solution for the final precipitation is unnecessary as sufficient 8-hydroxyquinoline is already in the solution. The addition of a wetting agent such as sulphonated lorol greatly facilitates the filtration of the aluminium quinolate by preventing the "creeping" of the precipitate which would otherwise occur.

The method has been tested on a series of synthetic solutions corresponding with a wide range of permanent magnet alloys, and it has been shown that under the best conditions an accuracy of  $\pm 0.2\%$  of the aluminium content is obtainable. Under routine working conditions, the tolerance, which may be defined as the acceptable conformity between the obtained result and the true value, is  $\pm 0.05\%$  aluminium on a result of 7% aluminium.

### Copper

The chemical determination of copper in the magnet alloy is complicated by the presence of the other alloying elements. For referee purposes<sup>1,3</sup> it is necessary to separate the copper by gassing the acid solution with hydrogen sulphide, and igniting the copper sulphide to cupric oxide. The determination can then be finished by several methods, of which the iodimetric titration is probably the most convenient.

The use of photometric methods is also subject to the necessity either to separate copper or to remove one or more interfering elements prior to the photometric determination. Thus, in the method of Haywood and Wood<sup>12</sup> which employs the diethyl dithiocarbamate complex of copper, it is necessary to make a preliminary separation of nickel with dimethylglyoxime.

For rapid routine analysis, a direct determination without a filtration is obviously desirable, and a polarographic procedure has been developed which makes this possible. The procedure is based on the work of Lingane and Kerlinger.<sup>9</sup> The method described by these authors cannot be directly applied to the analysis of the permanent magnet alloy, owing to the marked maxima which occur on the copper wave. These maxima are not eliminated by the addition of gelatine, as in the case of nickel. However, other maximum suppressors have been investigated, and it has been found that in the presence of the correct quantity of methyl cellulose, the maxima are suppressed and well-defined waves are obtained. (See Fig. 3(b)).

The finalised procedure for copper is as follows:—Pipette 25 mls. of the stock solution (as for cobalt) into a 100 ml. beaker and evaporate just to dryness. Take up in 2.0 mls. 12N hydrochloric acid, and transfer to a 100 mls. graduated flask with the aid of 60 mls. of water. Add 5.0 mls. pyridine and shake thoroughly. Add 5.0 mls. 2% methyl cellulose and dilute to the mark. Allow to settle somewhat, and pipette a portion of the supernatant liquid into a polarographic cell. Record the polarogram, after removal of oxygen as for nickel, using a voltage range of  $-0.1$  to  $-0.8$  and a galvanometer sensitivity of  $\frac{1}{4}$  to  $\frac{1}{16}$ , whichever is most suitable. Measure wave height and hence calculate the percentage of copper.

This method has been tested on a suitable series of standard solutions, and it has been shown to be sufficiently precise for the requirements of the analysis. Under routine conditions, experience has shown that the analytical tolerance is  $\pm 0.05\%$  copper on a result of 3%.

### Titanium

The photometric determination of titanium with hydrogen peroxide has been adopted for the routine analysis of the permanent magnet alloy. The interference due to the colour of ferric iron is masked by the addition of phosphoric acid, and it is an essential requirement of the method that the quantity of phosphoric acid used should be accurately measured, and that it should be added prior to the hydrogen peroxide. The presence of nickel, cobalt and copper in the alloy necessitates a blank absorptiometer reading and the determination of titanium by difference. It is not possible to carry out a direct reading, as in the case of cobalt, as the blank reading is rather large and varies too much with the composition of the alloy.

The method may be described as follows:—

Pipette two 25 ml. portions A and B of the main solution (as for cobalt) into a 100 ml. graduated flask. Add to A, 20 mls. of acid mixture (400 mls. phosphoric acid, 400 mls. 1:1 sulphuric acid and 200 mls. water), 5 mls. 3% hydrogen peroxide and water to the mark. Add to B, 20 mls. of acid mixture and water to the mark. Set Spekker Absorptiometer water-to-water 1.00 with 601 filters and take drum reading of the blank and colour solutions, using 1 cm. cells. Determine % titanium from the difference.

The standard graph, Fig. 4, is prepared from synthetic solutions corresponding to typical magnet alloy compositions.

This method is quite adequate for the routine examination of magnet alloys containing more than 0.5% titanium. It should be mentioned in this connection that interference from silicon which has been reported in photometric titanium determinations<sup>12</sup> has not been encountered in the present method.

However, for alloys containing less than 0.5% titanium, a significant portion of the titanium content may remain behind after the acid attack of the sample in the form of insoluble oxide, nitride or carbide. In such cases it is necessary to filter off any acid insoluble residue, fuse it with potassium bisulphate, and add the leached fusion to the main solution before determining titanium.

### Manganese

Manganese is very readily determined photometrically as permanganate. The method used for the analysis of the magnet alloys is well known and calls for no comment. It may be briefly described as follows:—

Pipette 50 mls. of the main solution (as for cobalt) into a 250 ml. beaker. Add 10 mls. phosphoric acid, 20 mls. 1:1 sulphuric acid and 0.3 gms. potassium periodate. Boil for 2 minutes and leave on the steam bath for a further 10 minutes. Cool and dilute to the mark in a 100 ml. graduated flask. Set Spekker Absorptiometer water-to-water 1.00 with 604 filters. Fill 1 cm. cell with solution and take drum reading. Add to the contents of the cell 2 drops of 2% sodium nitrite solution and stir until permanganate colour disappears. Take drum reading. Determine % manganese from difference of the drum readings.

<sup>12</sup> F. W. Haywood and A. A. R. Wood. "Metallurgical Analysis," Ad. Hilger, London, 1944.



## Silicon

The classical method of determining silicon in steel, by acid dehydration and subsequent ignition and weighing, can be readily applied to magnet alloys. The silica residue is rather badly contaminated with titania and alumina, and it is essential that the silica should be "fumed-off" with hydrofluoric acid, and the silicon content estimated from the loss in weight.

For batch analysis this gravimetric method requires a considerable amount of platinum ware, and a photometric method is to be preferred. The method of Vaughan<sup>13</sup> is not applicable to the magnet alloy, as 5% sulphuric acid will not dissolve the alloy in a reasonable time. However, the method of Gentry and Sherrington<sup>14</sup> permits a choice of solvents, and it is very suitable for the routine examination of the per-

total silicon content. In the case of the permanent magnet alloy, care must be taken that the samples used are not contaminated with moulding sand or large slag inclusions. Provided that this precaution is taken, the acid-insoluble portion of the alloy contains very little silicon, and, in fact, the results obtained by the photometric method are in excellent agreement with careful gravimetric analyses.

## Discussion

The chief feature of the procedures which have been developed for the routine analysis of the permanent magnet alloys is the use of instrumental rather than purely chemical methods for the determination of all but one of the constituents. It is claimed that such methods are faster than the chemical methods, require less skilled analysts, are as accurate as routine chemical methods and are less subject to operating-errors. However, it should be emphasised that the photometric determination of 30% cobalt or 20% nickel requires more attention to experimental detail than does the determination of 0.2% manganese or silicon, if results of acceptable precision are to be obtained. It is essential that careful attention should be paid to the operation of the Absorptiometer, and to the various factors which affect the formation of the coloured solution.

In the operation of the Absorptiometer thorough cleanliness is necessary. It is advisable to reserve separate cells for the determination of each element and to use the same cell for the preparation

of the calibration curve as for the determinations. A lens should be fitted to the instrument to permit accurate reading of the drum, and the drum should be used in a systematic manner to eliminate errors due to back-lash. If the lamp is removed for cleaning or for any other purpose, the standard curve should be checked at one point, and, if a new lamp is put in the instrument, a new calibration curve must be constructed. However, with reasonable care, the calibration curves are constant for several months. The use of nomograms connecting the weight of sample, the drum reading and the percentage of the element is recommended as they are easier to read than graphs and they save time spent on calculation.

In the preparation of the coloured solutions the reagents should be added accurately, and the solution well mixed before any further additions. Reagents are best added from automatic pipettes. Care must be taken that the reagents do not deteriorate or change their strength through evaporation.

An important source of error in photometric determinations which is often forgotten, is variation in the laboratory temperature. For the photometric determination of cobalt and nickel it is essential that the coloured solutions should be placed in a rough thermostat kept at say  $20^{\circ} \pm 2^{\circ} \text{C.}$ , for five minutes prior to reading on the Absorptiometer.

A valuable insurance against incorrect results is the running of a standard sample with every batch of

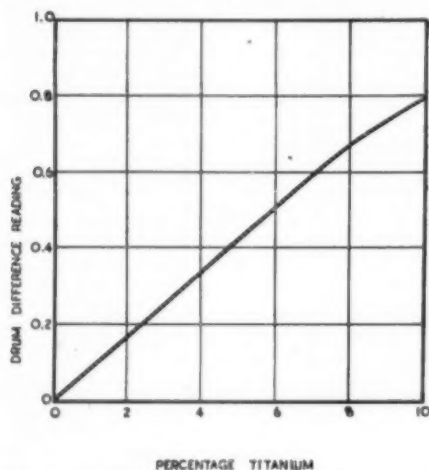


Fig. 4.—Calibration curve for titanium.

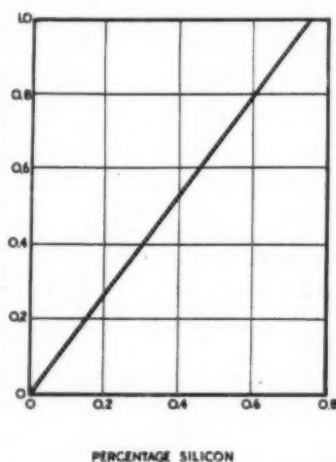


Fig. 5.—Calibration graph for silicon.

manent magnet material. The procedure which is suggested, may be briefly described thus:—

Weigh 0.25 gms. sample into a 350 ml. Erlenmeyer. Add 40 mls. 3N nitric acid and simmer until the sample dissolves. Dilute to 150 mls. with water and boil for 5 minutes. Cool and dilute to 500 mls. in a graduated flask. Pipette two 25 mls. portions (A and B) into dry 250 ml. beaker. Add to A from pipette 10.0 mls. 2.5% ammonium molybdate. Mix and stand for 5 minutes. Add 10.0 mls. 4% oxalic acid, mix and add 5.0 mls. 6% ferrous ammonium sulphate solution.

Add to B in this order with intermediate mixing, 10.0 mls. 4% oxalic acid, 10.0 mls. 2.5% ammonium molybdate, and 5.0 mls. 6% ferrous ammonium sulphate. Set Spekker Absorptiometer water-to-water 1.00 with 608 filters. Fill 2 cm. cell with colour solution A and blank B. Take drum readings and determine % silicon from difference.

The standard graph shown in Fig. 5 is identical with that used for simple steels. The photometric method is considerably faster than the gravimetric method, but it gives results of equal precision, and it has been found to be more reliable for routine control. It has been emphasised elsewhere<sup>14</sup> that a photometric method of this type determines only the "metallic silicon" content of a material, which may differ widely from the

13 E. J. Vaughan. "Further Advances in the Use of the Spekker Absorptiometer." Roy. Inst. of Chem. Monograph.

14 C. H. R. Gentry and L. G. Sherrington. *J. Soc. Chem. Ind.*, 1946, **65**, 90.

15 C. H. R. Gentry and G. P. Mitchell. In press. *J. Soc. Chem. Ind.*

material which is analysed. It suffices if a stock of standard solution is kept and suitable aliquots taken with each batch of nickel and cobalt determinations.

The procedures which have been described for the several constituents of the permanent magnet alloys permit the entire analysis, with the exception of silicon, to be made on one sample. The complete analysis as described, including silicon, can be carried out in less than three man hours per sample when done in batches. This time is very considerably less than that required to analyse this type of alloy by the classical methods of steel analysis.

Finally, it might be mentioned that the procedures which have been described are generally applicable to magnet alloys in the defined range. However, for certain types of permanent magnet material lying within much narrower composition ranges, the routine control of the manufacture is preferably made by spectrographic means. The spectrographic analysis which has been described in a separate paper,<sup>15</sup> is faster and more

convenient than the chemical method, but its application is more restricted.

### Conclusions

Procedures have been described for the determination of cobalt, nickel, aluminium, copper, titanium, silicon and manganese in permanent magnet alloys. The methods are, in general, modifications of known procedures, but, they permit the complete analysis of magnet alloys in much shorter time than if the analysis were made by the usual methods of steel analysis. The most significant feature of the method is the use of photometric and polarographic methods for the determination of elements in high percentages, with sufficient precision for the accurate control of the alloy composition.

The author wishes to thank Mr. L. G. Sherrington and Mr. G. Hutton for their co-operation in some of the work described, and Mr. J. A. M. van Moll and the directors of Philips Electrical Ltd. (formerly Philips Lamps Ltd.), for permission to publish this article.

## The Colorimetric Estimation of Tungsten in High Speed Steels Using Ammonium Thiocyanate and Titanous Chloride

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*A method is described whereby the advantages of the Spekker Absorptiometer may be applied to the estimation of tungsten in high speed steels.*

THE tungsten content of high speed steels is usually determined gravimetrically by a long and arduous method, the difficulties of which are well known. The tungstic acid precipitate is contaminated by chromium, iron, molybdenum and vanadium, and, in an accurate estimation these elements have to be determined and corrections made. Many laboratories also regard the precipitation as being incomplete unless the estimation has stood at least twelve hours even with the addition of cinchonine.

By using modifications of the methods recommended by Vaughan,<sup>1</sup> and Haywood and Wood,<sup>2</sup> manganese, chromium, vanadium, molybdenum, nickel and cobalt can now be estimated rapidly by use of the Spekker absorptiometer on a single weighing, in an overall time per sample of from two to three hours.

The following method in which the tungsten is determined on a fraction of the composite solution will result in a considerable saving of time and effort.

### Description of Method

#### Reagents Required.

##### Solvent Acid—

- Take 100 mls. distilled water.
- Add 12 mls. sulphuric acid. (S.G. 1.84).
- Add 60 mls. phosphoric acid. (S.G. 1.75).
- Use freshly mixed for each sample.

##### Nitric Acid—

(S.G. 1.20).

##### Hydrochloric Acid—

- Take 50 mls. hydrochloric acid. (S.G. 1.18).
- Add 50 mls. distilled water. Mix.

##### Titanous Chloride Solution—

- Take 30 mls. titanous chloride solution (15%).
- Add 30 mls. hydrochloric acid (S.G. 1.18).
- Boil, cool and make up to 500 mls. with 50% hydrochloric acid.
- Store in an inert atmosphere.

##### Acid Mixture—

- Weigh 150 gms. tartaric acid.
- Dissolve in 3% v/v sulphuric acid.
- Make to 1,000 mls. with 3% v/v sulphuric acid.

##### Ferric Chloride Solution—

- Weigh 3.75 gms. H.H.P. iron.
- Dissolve in 150 mls. 50% hydrochloric acid.
- Oxidise with potassium chlorate.
- Boil free from chlorine and make up to 2,000 mls. with distilled water.

##### Sodium Tungstate Solution—

- Weigh 8.965 gms. sodium tungstate (AnalaR).
- Dissolve, and make up to 1,000 mls. with distilled water.

##### Ammonium Thiocyanate Solution—

- Weigh 10 gms. ammonium thiocyanate (AnalaR).
- Dissolve in distilled water.
- Make to 100 mls. with distilled water.

##### Method.

- Weigh 2 gms. of the alloy into a 400 ml. beaker flask.
- Add the solvent acid mixture and simmer till reaction ceases.

<sup>1</sup> Monographs by Vaughan, E. J., M.Sc., F.R.I.C.

<sup>2</sup> Haywood, E. W., and Wood, A. A. R., "Metallurgical Analysis by Means of the Spekker Absorptiometer," Hilger, 1944.

Add cautiously, 10 mls. nitric acid (1.2) boil, fume, cool.

Dilute, boil for 3-5 mins., cool and make up to 200 mls. with distilled water.

Fractions of this composite solution can be used to determine the manganese, chromium, vanadium, molybdenum, nickel and cobalt content of the alloy by methods recommended by Messrs. Vaughan, Haywood and Wood.

For the tungsten<sup>3</sup> estimation:—

Take 50 mls. of the composite solution and make up to 100 mls. with distilled water. Mix.

Take 15 mls. of this solution.

Make up to 100 mls. with acid mixture. Mix.

Take 10 mls. of this solution.

Add 10 mls. of hydrochloric acid. (S.G. 1.18). Mix. Cool.

Add 10 mls. ammonium thiocyanate solution. Mix.

Add a volume of titanous chloride solution that is equal to that required to reduce 10 mls. of the ferric chloride solution, and make up to 50 mls. with 50% hydrochloric acid.

Mix and after 15 mins. determine the absorption of the yellow green colour on the Spekker Absorptiometer using the mercury vapour lamp, Ilford spectrum violet filters No. 601, and H503 heat absorbing filters, with a water setting of 1.20.

For the success of this method great care should be taken in the pipetting, and a careful check should be kept on the reducing power of the titanous chloride solution.

Molybdenum does not interfere,<sup>4</sup> the reddish brown molybdenum complex is produced initially but rapidly fades.

#### Graph.

1 ml. of the sodium tungstate solution contains 0.005 gm. tungsten.

Weigh ten samples of 0.5 gm. H.H.P. iron.

Add to each one quarter the volume of solvent acid that is used for the preparation of the composite solution.

Add immediately; from 1 to 20 mls. of the sodium tungstate solution giving an equivalent range of from 1 to 20% tungsten.

Simmer until reaction ceases.

Add cautiously, 5 mls. nitric acid (S.G. 1.2), boil, fume, cool.

Dilute, boil for 3-5 mins. cool and make up to 100 mls. with distilled water.

Take 15 mls. each of these solutions and proceed as in the composite method.

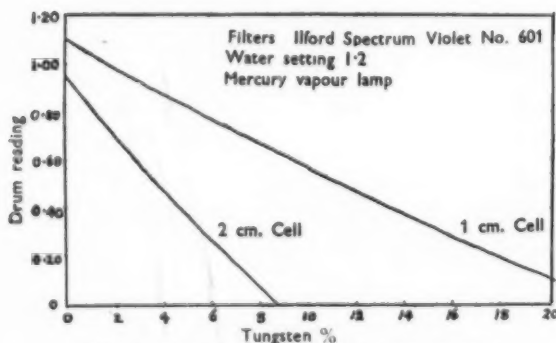


Fig. 1.—Calibration graph for tungsten.

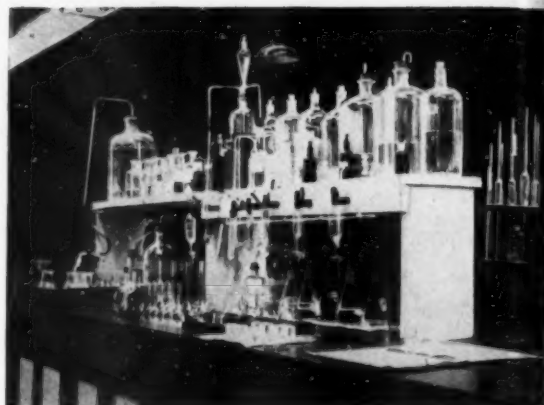


Fig. 2.—Bench on which Spekker work is prepared. Comparison With Gravimetric Results

A number of samples were taken having varying tungsten content, and a comparison made of the results estimated, both gravimetrically and colorimetrically. (Table 1).

A good agreement was obtained, though it was noted that, on the average, higher results were obtained colorimetrically.

TABLE I

Alloy	Spekker % W	Gravimetric % W	Alloy	Spekker % W	Gravimetric % W
1 .. ..	1.42	1.43	5	16.12	16.00
2 .. ..	3.60	3.60	6	17.91	17.94
3 .. ..	6.60	6.53	7	18.39	18.24
4 .. ..	13.22	13.37	8	20.16	20.02

#### Reproducibility

The reproducibility was tested on samples of B.C.S. W<sub>2</sub> and 220 alloy steels (Table 2). The results given are those obtained by different chemists over a period of some weeks, and show a variation that compares favourably with the analyses given in the B.C.S. analysis sheets.

TABLE II

Alloy	B.C.S. % W	Spekker % W					
W <sub>2</sub> .. ..	16.12	16.20	16.20	16.12	16.17	16.20	16.17
220 .. ..	6.74	6.84	6.82	6.80	6.73	6.83	6.77

Thanks are due to the management of Messrs. Guest, Keen and Nettlefolds, Ltd., for permission to publish this article and to Mr. T. W. Whiting, Chief Metallurgist, for his advice and encouragement.

<sup>3</sup> Snell, F. D., and Snell, C. T., "Colorimetric Methods of Analysis," 1944, Vol. 1, 378.

<sup>4</sup> Hurl, L. C., and Allen, H. O., *Ind. Eng. Chem., Anal. Ed.*, 1935, 7, 396.

#### Iron and Steel Distribution Booklet

"Notes to Consumers," a booklet covering various aspects of the Iron and Steel Distribution Scheme, including the names and addresses of authorising Departments with details of their authorising responsibilities and the items that are covered by direct authorisations, will be on sale at His Majesty's Stationery Office shops as from June 1st, 1948, price 6d.



# MICROANALYSIS

CHEMICAL AND PHYSICAL METHODS

APPARATUS

METALLURGICAL

APPLICATIONS

TECHNIQUE

THE discussions which have been taking place in our American contemporary "Analytical Chemistry," on the nature of analytical chemistry and the role of the analytical chemist provide continuing food for thought, and must engage the minds of all concerned with these problems for many months to come. Almost every one of the distinguished contributors to this feature makes points on which one would like to expand, or with which one would wish to argue. The mere fact that one is not in wholehearted agreement with everything that has been printed is an excellent sign both of the importance of a full discussion, and of the aliveness of the topic. Previously we have mentioned points with which we are in agreement. We would like, just for a moment, to turn to a statement which seems to us less calculated to meet with everyone's approval. Discussing the need for many types of chemists and physicists to cope with the great expansion of analytical chemistry on the instrumental side, W. C. McCrone comments that "it is essential to emphasise that the best 'analyst' in such a group may not know what happens when sodium bicarbonate and hydrochloric acid are mixed." This, if true, brings us back to the essential distinction between the analyst and the analytical chemist—the former merely a technician as against the fundamental chemical training of the latter. It seems to us that McCrone has not made this distinction in his remarks, in which he seems to consider only the technical experts, without a proper recognition of the necessity for the guiding hand or mind behind the hand that actually turns the dials. It is, of course, best when the analytical chemist is skilled in the use of the machine which he uses. Failing this, he must be skilled in deciding which machine to use, and what its limitations may be. To eulogise the "analyst," and to forget to consider the role of the "analytical chemist" is to make the dangerous assumption that the Chief Engineer is responsible for the safe arrival of the liner, and that the Captain, on the bridge, is merely an ornament.

## Group Separations in Inorganic Qualitative Analysis

### Part I—Schemes Based on Sulphuretted Hydrogen

By Francis R. M. McDonnell and Cecil L. Wilson

*Microchemistry Laboratory, Chemistry Department, The Queen's University of Belfast).*

REASONABLY well formulated schemes of analysis were apparently in existence as early as 1800, but the foundations of classical inorganic qualitative analysis may be said to have been laid by Fresenius who, in the year 1840, as a student at the University of Bonn, published the first edition of his well-known textbook.<sup>1</sup> In this there was presented a selection from, and a refinement of, previous schemes, and the scheme so arrived at, with variations, is now so well known, and so widely used, that it is usually referred to as the classical scheme.

In it, as is well known, the commoner cations are divided into five groups, as shown in Table I.

The original Group III is now often precipitated in two parts, the first sub-division, precipitated by ammonium hydroxide in the presence of ammonium chloride, containing the iron, aluminium and chromium cations as

TABLE I.

Group	Precipitant	Ions Precipitated	Conditions
I	HCl	$\text{Ag}^+$ , $\text{Hg}_2^{++}$ , $\text{Pb}^{++}$	Chlorides
II	$\text{H}_2\text{S}$	$\text{Hg}^{++}$ , $\text{Cu}^{++}$ , $\text{Bi}^{+++}$ , $\text{Pb}^{++}$ , $\text{Cd}^{++}$ ; $\text{Sb}^{+++}$ , $\text{Sn}^{++}$ , $\text{Sn}^{++++}$ , $\text{As}^{+++}$	Sulphides insoluble in acid solution.
III	$(\text{NH}_4)_2\text{S}_7$	$\text{Fe}^{+++}$ , $\text{Al}^{+++}$ , $\text{Cr}^{+++}$ ; $\text{Mn}^{++}$ , $\text{Co}^{++}$ , $\text{Ni}^{++}$ , $\text{Zn}^{++}$	Sulphides insoluble in alkaline solution.
IV	$(\text{NH}_4)_2\text{CO}_3$	$\text{Ca}^{++}$ , $\text{Sr}^{++}$ , $\text{Ba}^{++}$	Carbonates
V	—	$\text{Mg}^{++}$ , $\text{Na}^+$ , $\text{K}^+$	Remain in solution.

hydroxide, while the remaining sulphides are then precipitated by ammonium sulphide.

<sup>1</sup> Fresenius, "Qualitative Analysis," 1840.

## Modifications to Groups I and II

From time to time, various modifications, of greater or less degree have been proposed to this basic scheme. On examining these in detail, the first point which emerges is that very few proposals to replace the precipitation of the first group chlorides have been put forward. Likewise, the precipitation of sulphides in the second group has been retained widely, although a number of alternative means of producing the precipitates have been suggested.

Hall<sup>2</sup> suggested that red phosphorus should be used as the first precipitant. This reagent places both forms of mercury in the first group, and leaves the whole of the lead to be precipitated in Group II. Hall also advocated thioacetic acid or its ammonium salt as the precipitant for the sulphides of the acid group. An extra group was formed by the precipitation of potassium with phosphotungstic acid.

Rosenstein<sup>3</sup> has also suggested the use of red phosphorus. His scheme, shown in outline in Table II, commences by boiling a nitric or sulphuric acid solution of the cations with red phosphorus, thus precipitating a number of ions as metals or phosphides. The filtrate receives treatment according to the normal manner, certain simplifications being found. Thus copper and mercuric sulphides are removed from the sulphide group, while it is claimed that the lower valence forms produced by the treatment should simplify the analysis of Group III. As far as can be determined, this scheme is based on Rosenstein's examination of the behaviour of ions with red phosphorus, and has not received any extensive practical tests.

TABLE II.

To a dilute HNO <sub>3</sub> or H <sub>2</sub> SO <sub>4</sub> solution add red phosphorus in excess, and boil for 30 minutes.	
<b>Residue</b> Ag, Cu, Hg <sub>2</sub> , Au, Pd, Os, Se as metals or phosphides. Dissolve by repeated boiling with aqua regia, adding KClO <sub>4</sub> if necessary. Evaporate, and take up in HNO <sub>3</sub> .	<b>Filtrate</b> Analysed with H <sub>2</sub> S, etc., as in classical scheme.
<b>Residue</b> AgCl	<b>Filtrate</b> Cu, Hg, Au, Pd, Os, Se

Tananaev<sup>4</sup> as shown in Table III, precipitates Groups I, II and III as sulphides, and achieves a separation of these by treatment of the precipitate with acids of varying concentrations.

TABLE III.

From ammoniacal solution precipitate the cations whose sulphides are insoluble in alkaline solution. Heat the residue with excess acetic acid.

<b>Residue</b> Neutralise with NaOH and add Na <sub>2</sub> O <sub>2</sub>	<b>Filtrate</b> Al, Fe, Co, Ni, Mn
<b>Residue</b> Wash. Treat with cold 0.6 N HCl	<b>Filtrate</b> Cr
<b>Residue</b> Boil with 4 N HCl	<b>Filtrate</b> Zn, Pb, Cd
<b>Residue</b> Treat with aqua regia	<b>Filtrate</b> Bi
<b>Residue</b> AgCl	<b>Filtrate</b> Hg, Cu

Gelback,<sup>5</sup> also, precipitates Groups I and II as sulphides, using sodium sulphide. The insoluble sulphides are tested for in the usual manner after first testing for silver with hydrochloric acid.

## Variations to Later Groups

In the analysis of the filtrate from Group II, a large number of variations have been put forward. Tomsich and Carney<sup>6</sup> have presented a method for precipitating aluminium, ferric iron and chromium as complex benzoates. This reaction is carried out in acetic acid solution, in the presence of ammonium chloride, by boiling with 10% ammonium benzoate solution, when the complex benzoates of the above metals are precipitated. The filtrate from this treatment is then analysed in the usual manner. The authors put forward several claims for their method. It offers a complete separation of the trivalent and divalent cations in this Group. Since the solution is fairly strongly acid (pH = 3.8) there is no danger of the partial precipitation of manganese as hydroxide or oxyhydroxide. The precipitate is easily coagulated, and the slimy precipitates, difficult to filter, which are obtained in ammonia precipitates, are thus avoided.

Ostroumov and Bonschtein,<sup>7</sup> to overcome the difficulty of the absorption of the alkali cations on the hydroxide precipitate of Group III, suggest the use of hexamethylenetetramine. The precipitation is carried out in the presence of ammonium chloride at an initial temperature of 60°C. After the addition of the reagent, hydrogen sulphide is passed through the solution at a slowly rising temperature for an hour. The pH gradually rises, and each sulphide is precipitated more or less separately, forming granular precipitates which do not absorb calcium, magnesium or alkali metal ions. If nickel or cobalt is present, the addition of a little pyridine hydrochloride, before the hexamethylenetetramine, is advised.

Several workers have evolved schemes for the analysis of the classical Groups III and IV depending on the use of the phosphate ion. Remy<sup>8</sup> has proposed such a scheme. After removal of hydrogen sulphide, as shown in Table IV, the filtrate from Group II is nearly neutralised with sodium carbonate, and then poured into

TABLE IV.

Groups I and II are precipitated as usual. Boil off H<sub>2</sub>S, add a few drops of concentrated HNO<sub>3</sub>, boil, neutralise with ammonium carbonate until the precipitate which forms is just redissolved. Pour, with vigorous stirring, into an equal volume of moderately warm NaCH<sub>3</sub>, and add excess (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub>.

<b>Residue</b> Dissolve in a small amount of HCl. Add K <sub>2</sub> SO <sub>4</sub> .	<b>Filtrate</b> Add ammonium sulphide.
<b>Residue</b> CaSO <sub>4</sub> , SrSO <sub>4</sub> , BaSO <sub>4</sub>	<b>Filtrate</b> Boil with solid sodium acetate and ammonium chloride
<b>Residue</b> ZnS, CoS, NiS	<b>Filtrate</b> Alkali metals
<b>Residue</b> Dissolve in HCl. Nearly neutralise with sodium carbonate and pour into warm H <sub>2</sub> O <sub>2</sub> and 20% NaOH	<b>Filtrate</b> Add ammonium oxalate
<b>Residue</b> Fe(OH) <sub>3</sub> , Mn(OH) <sub>2</sub>	<b>Filtrate</b> Add NH <sub>4</sub> Cl
<b>Residue</b> Al(OH) <sub>3</sub>	<b>Filtrate</b> Cr
<b>Residue</b> Ca Add NH <sub>4</sub> OH	<b>Filtrate</b> Mg(NH <sub>4</sub> )PO <sub>4</sub>

<sup>2</sup> Hall, *Trans. Maryland Acad. Sci.*, 1921, **4**, 8; *C.A.*, 1921, **15**, 1667.

<sup>3</sup> Rosenstein, *J. Amer. Chem. Soc.*, 1920, **42**, 883.

<sup>4</sup> Tananaev, *Z. anorg. allgem. Chem.*, 1924, **140**, 320; *C.A.*, 1925, **19**, 1108.

<sup>5</sup> Gelbach, *J. Chem. Educ.*, 1933, **10**, 621.

<sup>6</sup> Tomsich and Carney, *ibid.*, 1940, **17**, 29.

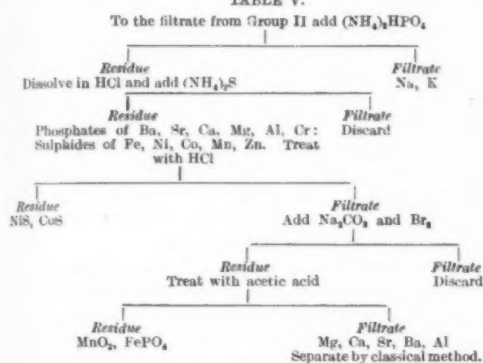
<sup>7</sup> Ostroumov and Bonschtein, *Zar. Lab.*, 1940, **9**, 139; *C.A.*, 1940, **34**, 5777.

<sup>8</sup> Remy, *Z. anal. Chem.*, 1919, **58**, 385; *B.C.A.*, 1920, **118**, ii, 186.

moderately warm sodium hydroxide and ammonium phosphate solution. This leaves zinc, cobalt, nickel and the alkali metals in solution. The residue, dissolved in warm hydrochloric acid, is treated with sulphate to remove the alkaline earth metals. The filtrate is then treated with sodium acetate and ammonium chloride, precipitating iron, manganese, aluminium and chromium, while magnesium, and any calcium not precipitated as sulphate, remain in solution.

A somewhat similar scheme has been put forward by Petraschenj.<sup>9</sup> In this case (Table V), after the removal of hydrogen sulphide, the addition of ammonium phosphate precipitates all the remaining cations with the

TABLE V.

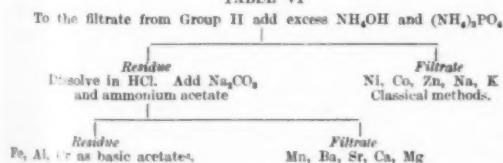


exception of sodium and potassium. The precipitate is dissolved in hydrochloric acid, and ammonium sulphide added, precipitating the complex mixture of sulphides and phosphates. From these, in turn, nickel, cobalt, manganese and iron are removed, and the remaining ions, in solution, are subjected to normal methods of separation.

Austin<sup>10</sup> first removes barium and strontium from the hydrogen sulphide filtrate, made 0.2N with respect to HCl, a sulphates. Iron, aluminium and chromium are then precipitated as phosphates at pH 3.2-3.4. The solution is then buffered to pH 4.6-4.8, when the action of sulphuretted hydrogen precipitates zinc, nickel and cobalt. Excess of the gas is removed, manganese is precipitated as the dioxide by oxidation with sodium hypochlorite, calcium as the oxalate, and magnesium as the phosphate.

Kreschkov<sup>11</sup> adds ammonia and ammonium phosphate to the Group II filtrate. As shown in Table VI, this leaves nickel, cobalt, zinc, sodium and potassium in solution. The residue is dissolved in HCl, and iron, aluminium and chromium are removed as basic acetates while manganese, barium, strontium, calcium and magnesium remain in solution.

TABLE VI



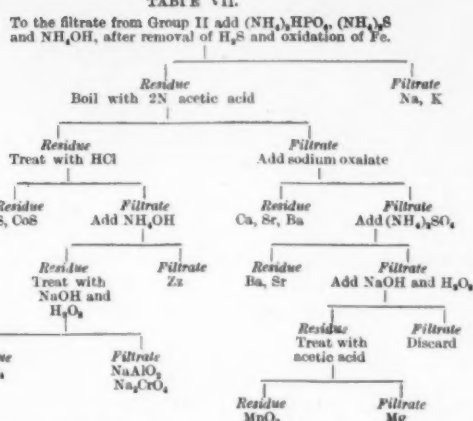
Scheinkman<sup>12</sup> first precipitates all cations but those of the alkali metals from the Group II filtrate (Table VII).

<sup>9</sup> Petraschenj, *Z. anal. Chem.*, 1936, **106**, 330; *C.A.*, 1937, **31**, 62.

<sup>10</sup> Austin, *Analyst*, 1940, **65**, 335.

<sup>11</sup> Kreschkov, *J. Gen. Chem. Russ.*, 1932, **2**, 593; *B.C.A.*, 1933, 246.

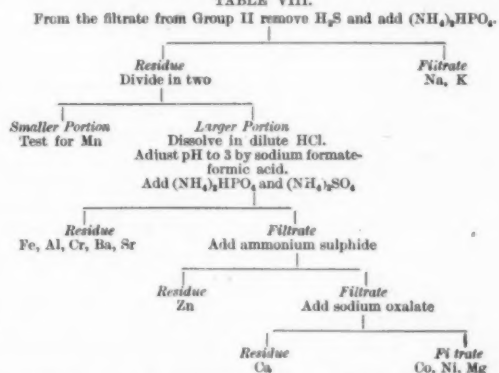
TABLE VII.



The residue is boiled with acetic acid, leaving aluminium, iron and chromium phosphates and the sulphides of nickel, cobalt and zinc undissolved. Subsequent separations are achieved with hydrochloric acid, ammonia, sodium hydroxide and hydrogen peroxide. In the original acetic acid extract, the alkaline earth metals are separated as oxalates and sulphates. Manganese and magnesium are precipitated with sodium hydroxide and hydrogen peroxide.

In a method designed to obviate the loss of certain ions, and to retain all the ions in their original relative proportions, Smith<sup>13</sup> removes all the insoluble phosphates from the Group II filtrate. As shown in Table VIII, a small portion of the precipitate is then tested for manganese with sodium bismuthate. The remainder is dissolved in dilute hydrochloric acid, and the pH is adjusted by buffering to pH 3. Iron, aluminium and chromium are removed as phosphates, and barium and strontium as sulphates. The latter are subsequently

TABLE VIII.



converted to carbonates. Zinc is then precipitated as the sulphide and calcium as the oxalate. In the filtrate, cobalt, nickel and magnesium are identified by tests with organic reagents, without further separation.

Palmieri<sup>14</sup> precipitates all the remaining cations except the alkalis from the filtrate from Group II. The precipitate is dissolved in acid, and the alkaline earth cations removed with sulphuric acid and ethanol. Otherwise the normal procedure is followed.

<sup>12</sup> Scheinkman, *Z. anal. Chem.*, 1933, **91**, 415; *C.A.*, 1933, **27**, 1841.

<sup>13</sup> Smith, *J. C. S.*, 1933, 253.

<sup>14</sup> Palmieri, *Officiana*, 1932, **5**, 136; *B.C.A.*, 1933, 584.

To be continued



# Miscellaneous Microchemical Devices

## XIII—A Micro-Mortar for Powdering Solids

By J. T. Stock and M. A. Fill.

*Chemistry Dept., L.C.C. Norwood Technical Institute.*

THIS device, which may be simply and rapidly constructed in a variety of sizes, is made from an ordinary pyrex test tube or boiling tube. A peripheral scratch having been made round the tube at a distance from the closed end of about one and a half to twice the diameter, the end is cut off by means of an electrically-heated nichrome wire<sup>1</sup>. The rim of the cup thus formed is fire-polished and a lip is formed to retain the pestle, as shown in Fig. 1. The construction of the pyrex pestle is self-evident. Before use, the pestle is ground into the mortar with the finest emery powder and water; all traces of abrasive are then thoroughly washed out.



0 5 CM

Two sizes of micro-mortar.

Although simple, the mortar is very effective. For the purposes of handling, a small block of wood or a large cork, in which a hole of suitable dimensions has been bored, is suitable. If a simple wire or glass supporting cradle is constructed, the whole unit in its smaller sizes is light enough to be carried on a microbalance, so that materials may be weighed directly into the mortar.

<sup>1</sup> A. J. Ansley, *Jour. Sci. Instr.*, 1936, **13**, 194.

## XIV—A Hydrogen Sulphide Generator for Microanalysis

By J. T. Stock and M. A. Fill.

THE device (Fig. 1) consists of two parts, the generator proper and an electrically-operated pump which delivers the gas under suitable pressure. As regards the generator portion, a short, stout test tube (7 cm.  $\times$  2.5 cm.) in the bottom of which a 5 cm. length of 4 mm. bore glass tubing has been sealed as shown, serves as the container for iron sulphide B. To prevent the latter from blocking the tubing, a few pieces of broken glass are inserted before introducing the charge. A rubber stopper carrying an "L"-shaped exit tube closes the mouth of the container; the upright arm of the "L" is about 10 cm. long. A small beaker containing a little hydrochloric acid A supports the container.

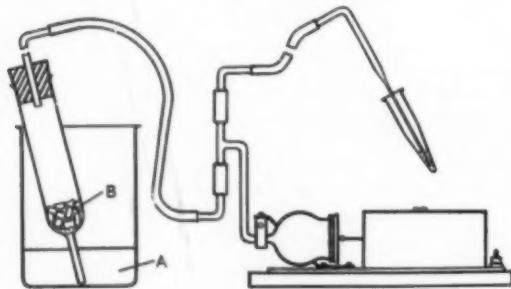


Fig. 1.—Hydrogen sulphide micro-apparatus.

The pump (Fig. 2), which is described fully elsewhere<sup>1</sup>, is driven by an electric bell movement, which is so arranged that the hammer depresses rubber diaphragm A stretched over the mouth of the adapted thistle funnel. At each stroke, gas is forced out through valve B, while the return of the diaphragm causes gas to be drawn in through valve C (both valves are discs of thin rubber). The rapidly repeating cycle of operations gives an apparently continuous stream of bubbles.

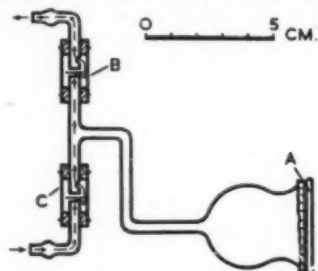


Fig. 2. Pump unit.

It should be noted that the generation of gas occurs under reduced pressure, and cannot commence until the starting of the pump draws the acid into the container. As in the case of the ordinary Kipp generator, the acid recedes from the iron sulphide when the rate of gas generation exceeds the rate of usage. Since the pump does not produce a suction greater than about 12 cm. of water, there is no danger of acid being drawn into the pump.

<sup>1</sup> J. T. Stock and M. A. Fill, *Analyst*. (In the press.)

## Gas Turbine School

THE first of nine courses to be held this year at Britain's School of Gas Turbine Technology, Lutterworth, commenced on May 31st, when technicians from various commercial organisations attended for three weeks' instruction in the industrial uses of the gas turbine. In addition to engineers from the United Kingdom, Dominion representatives have also enrolled for the course.

Responsibility for the School and its syllabus rests with Power Jets (Research and Development) Ltd., a Company owned by the Ministry of Supply. Having learnt the basic theory of the gas turbine, the "students" are shown practical testing procedure and instrumentation. Highlight of the course is a series of lectures given by specialists in subjects such as fuel problems, application in locomotives and marine installation, metallurgy and combustion components.

So far, 800 British and 35 foreign engineers have passed through this institution, the only one of its kind in the world. It is the Company's ultimate intention to cater for student engineers with courses during the normal holiday periods and also to hold practical classes for such men as testers and engine fitters. The courses have been divided into four distinct categories—industrial, aero-engine, draughtsmen's and international. The international course will be held in August and will be a combination of the industrial and aero-engine syllabuses.

THE telephone number of the Head Office and London Sales Office of Northern Aluminium Company, Ltd., is changed to Temple Bar 8430.

# METALLURGICAL DIGEST

## Production Electroforming

By Thomas S. Blair

**E**LECTROFORMING has simplified many design problems in the past, primarily in small lot, precision jobs. Only recently have improved techniques permitted its use on a mass production scale to allow freer design and cheaper production of many articles.

Electroforming involves the electro-deposition of a relatively thick layer of metal on a mould or matrix and subsequent removal of the shell from the mould so that a self-supporting part or formed article is produced. The process has been fairly widely used for some time in the manufacture of dies for moulding plastics, in making phonograph record masters and stamping plates, electrotypes, pitot tubes, radar wave guides, venturi nozzles, and in similar applications where precision forming of the article has been essential.

These uses, however, have been for the production of a comparatively limited number of pieces in which close tolerances or difficult shapes were required, and it is only recently that adaptation and improvement of techniques have brought the process into production usage.

Copper, silver, iron and other metals can be and are electroformed, but nickel, because of its hardness, tarnish resistance and comparatively rapid plating rate has been the standby in production work. Using a Watts bath at 150° F. with modified operation techniques, and current densities between 200 and 250 amp. per sq. ft., Camin Laboratories, Inc. plates nickel at a rate of 0.010 in. per hr., and it is this speed which enables the method to be commercially competitive. Actually at least double this plating rate is possible and is used when forming moulds or pieces in which only internal surfaces next to the mandrel are critical. However, with economically feasible bath agitation, general forming

at more than 0.010 in. per hr. is unsuccessful as the external surfaces tend to develop ripples and irregularities.

The process, as employed by Camin, involves fixing the mandrels on which the articles are deposited to an endless moving conveyor which spins and plunges them as it moves through the 38-ft. bath length. The conveyor speed is controlled so as to determine the plating time and thereby the thickness of the piece. Automatic control devices continually circulate and filter the plating solution and assure proper bath temperature and current density.

Although electroforming can compete favourably with a simple drawing operation, the really substantial savings arise in the realm of high design or special shaping where irregular patterns ordinarily requiring a series of forming steps are just as simple to make in the original electroforming operation as a perfectly plain shape.

For the comparatively simple shapes, in which the mandrel can pull out of the plated form, a hardened alloy steel mandrel is used to which a thin layer of a parting medium has been applied. Parting mediums, such as carbon black, beeswax, collodion, turpentine, potassium dichromate and solutions of selenium or arsenic salts, allow plating over the mandrel but prevent a strong bond from forming between the mandrel and the plated metal, so that after completion of the plating operation friction applied to the spinning mandrel on the conveyor frees the plated shape.

There are other means for subsequent freeing of the shell. The mandrel can be evenly coated with tin either by a plating operation or by a dip and wipe. Then immersion of the mandrel with its plated shell in a hot oil bath will melt the tin and separate the two pieces. A third alternative is to choose a mandrel of glass or some material having a correspondingly low co-

efficient of expansion. On heating, the comparatively greater expansion of the shell will part the pieces.

In forming complicated parts that involve undercuts, fusible mandrels made of a bismuth-lead eutectic alloy are used. These mandrels are centrifugally cast in the desired shape, the part is formed around it and the mandrels melted out in a hot oil bath.

An advantage of the electroforming method is its precision. With the permanent mandrels tolerances of  $\pm 0.0005$  in. are no problem as the mandrels can be machined and ground to a very precise form to which the plating layer will absolutely conform internally. Thickness and outside tolerances of the part are controlled by adjustment of the plating bath characteristics. Tolerances of  $\pm 0.001$  in. on external dimensions are easily held in production.

The mandrel is reproduced with exceptional faithfulness and precision. Engravers' marks, perhaps 0.00002 in. wide, and the minute sound groove configurations of phonograph record masters are exactly duplicated.

Thin wall and capillary tubing are easily and accurately formed. Nickel tubing with a 0.005-in. wall becomes quite expensive when formed by standard drawing and annealing operations, but by electroforming it can be formed relatively cheaply and with high precision. Capillary with a 0.002 or even 0.001-in. wall thickness is easily produced over a wire mandrel.

Another advantage of electroformed work is the high internal polish of the pieces, requiring no subsequent finishing operations. The mandrel surface must be scrupulously clean and polished and this reflects in the formed piece when it is removed from the mandrel. In small tubing to be used for sleeve-type bearing applications, such as in the second hand workings of a watch or clock, this internal polish is very desirable.

Electrodeposited nickel is quite fine grained, metallurgically sound, strain free and is said to be stronger and harder than nickel which has been cold worked and annealed.

From *The Iron Age*, 1948, 161, April 1, 72-75.

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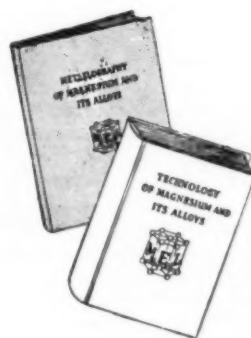
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# ELEKTRON

## MAGNESIUM ALLOYS



Electrodeposition on plastics is another operation being done on a production basis. Phenolics, polystyrene, ureas and other thermosetting, thermoplastic and non-plasticised types which are permanent at 150° F. can be readily metallised. The plastic

articles are roughened and sensitised and a very thin pure silver film is imparted to the piece by chemical reduction. Nickel is then electroformed on silver to desired thickness and a secondary finish of chrome, gold or metal desired can be flashed on top of nickel.

## Metal Coatings by High Vacuum Evaporation

By Philip Godley

DURING recent years the scope of the evaporation process as a means of producing thin metal coatings has been greatly increased by a better understanding of high vacuum engineering and the development of large oil diffusion pumps. Briefly the process consists of condensing metal vapour on the surface of the article to be coated. In order to obtain high rates of evaporation it is necessary to achieve high temperatures and low pressures in the evaporator in which the process is carried out. As a comparison, the temperature at which the vapour pressure reaches 0.01 mm. of mercury is 1,000° C. for aluminium, 270° C. for cadmium, 1,280° C. for chromium, 1,460° C. for gold, 1,050° C. for silver and 3,200° C. for tungsten. The low pressures employed reduce the temperature to which the metal must be raised and reduce the risk of chemical reaction with the atmosphere and it is for the latter reason that such low pressures as  $10^{-4}$  to  $10^{-3}$  mm. Hg. are used. Should collision of particles of metal occur, due to too high a pressure, particles of greater than molecular dimension will be deposited and a dull off-colour coating will result. The article to be coated is usually at a much lower temperature than the evaporating source and condensation readily occurs.

The equipment consists of an evaporator tank, which for large parts or batches of small parts may be 4 ft. in diameter and 4 ft. long, connected to one or more oil diffusion pumps backed by mechanical pumps. Separate roughing pumps are used for the preliminary stages of evacuation. The tank contains supports for the articles to be coated, which are usually placed above the source, and anything from a few inches to a few feet away from it. The metal vapour source may be one of the following: (1) A tungsten coil with pieces of the metal wire attached at intervals and evaporated by the passage of current through the

coil. Aluminium is evaporated in this way. (2) A refractory metal boat containing the metal has a heavy current passed through it. Gold, silver and copper are examples of the metals evaporated in this way. (3) A ceramic crucible containing the metal may be heated by a resistance wire wrapped round it or by an H.F. induction coil. The tank also contains a means of degassing the article and the tank walls. For this purpose an electrode inserted through the base plate is supplied from a high voltage transformer and the ions created by the glow discharge bombard the articles and contribute energy to the evaporation of the absorbed layer during the roughing-down of the tank.

Thin films are liable to peel off and in any case it is not economically practical to deposit films thicker than 0.001 mm. In the case of flexible materials there is no stiffening caused by the evaporated film. Since they are so thin, it is not possible to polish the coatings, but even so aluminium and silver coatings reflect well over 80% of the incident light. It is generally agreed that the films consist of agglomerates formed by migration of atoms due to surface forces after condensation. Most films contain some oxide of the evaporated metal the concentration being greatest in the initial layer, apart from whatever forms on the surface upon exposure to the atmosphere. The films show much higher electrical resistance than the massive metal and also act as a partial barrier to water vapour but they are generally used for their optical properties.

The uses of evaporated coatings include television mirrors, astronomical telescope mirrors and parts of the interior of light bulbs. Quartz crystals have been coated with gold to load them to respond to definite frequencies and with silver for soldering leads to them. Thin plastic sheeting may be coated for use in making decorative wrappings and condensers may be

built up from coated paper. In the latter connection, should an arc occur through the paper, the metal will burn away and leave an insulating area which extinguishes the arc.

Until recently the method was almost entirely restricted to batch operation but pilot plants are now in operation which can coat 5,000-6,000-ft. rolls of flexible material with aluminium, silver, copper or gold in a few hours and another will coat 5,000-ft. rolls of condenser paper with zinc in a few minutes.

## Carbide Inserts for Single Point Tools

By THOMAS E. LLOYD

CONSIDERABLE interest is being displayed in the U.S.A. in a new type of single point tool in which the cutting component is a length of carbide held mechanically in the tool holder. The carbide insert may be round, triangular or square and a new cutting edge may be obtained by turning the insert. The tool holder is so designed that besides holding the insert in position, the latter can be adjusted in and out of the holder by means of a set screw placed directly under and in line with the insert.

The advantages claimed are: (1) The ease of insertion of the tool in the holder; (2) The ability to present a new cutting edge without regrinding the tool; (3) The elimination of the need for certain critical tool angles required by standard carbide tipped tools; (4) The long tool life made possible by the length of the insert; and (5) The long tool life per grind.

Whilst there is little in the way of concrete information on the advantages and disadvantages, General Motors claim that, in turning transmission parts, round carbide inserts will cut up to 12 times as much per grind as standard carbide tools and can be ground up to 12 times as often as standard tools before being discarded. Most experimental work has been done with the round insert ground flat on the top and anchored securely in position, but the Heald Machine Co. has done some boring work with the insert floating free in the toolholder and it has been found that in boring the insert rotates as boring progresses, so that no one arc segment of the edge bears the total brunt of the cutting and as a result the tool may remain cooler.

One difficulty encountered with boring, skiving and turning with these tools is, unless an interrupted cut is being made there is no way to break up the chips.

From *Iron Age*, 161, April 15, 1948, 82-83.

From *Iron Age*, 1948, 161, April 1, 90-94.

# Powder Metallurgy as Applied to Permanent Magnets

By A. E. Franks

AS the available high-quality permanent magnet alloys are extremely hard and brittle they cannot be machined to shape and finishing and sizing is possible only by grinding. Cast magnets are readily made in the larger sizes but difficulties were encountered in making small magnets of various shapes by casting. Powder metallurgical methods were tried successfully and a working installation was in operation in 1936. Approximately 12 permanent magnet alloys can now be manufactured by powder metallurgy techniques, the process consisting of pressing to shape, sintering and heat treating to develop the required characteristics.

As the magnetic properties are extremely sensitive to small variations of composition and microstructure, close control of the operations is necessary. All powder is checked for (1) Chemical analysis; (2) Maximum particle size; (3) Particle size distribution; (4) Shape and hardness of particles; (5) Flow characteristics; and (6) Compression ratio. Pure metal powders are usually from 99-99.99% pure. For most purposes the powder should pass through a 100-mesh screen, i.e., must have a maximum dimension of about 149 microns. Samples of powder are passed through a standard set of graded screens the portion passing through each being weighed. An example of particle size distribution is as follows: + 100 mesh, nil; - 100 + 200 mesh, 0-10%; - 200 + 325 mesh, 5-15%; and - 325 mesh, 75-95%. Special powders with a maximum particle size of 4 microns have been used, but in general the cost is not justified by the improvement

obtained. Soft spherical particles are considered to exhibit the best pressing characteristics.

Batches of powder are mixed in the desired proportions in a ball mill and a small quantity of lubricant—usually a metal salt of a fatty acid or a hydrogenated oil—is added to the premixed powders and further mixing causes each particle to be coated with a film of lubricant. By reducing the friction between particles and between the powder and the die wall, a more uniform pressure distribution in the pressed parts is attained and this results in a more uniform shrinkage on sintering. Samples from each batch of mixed powder are processed and tested before approval of the powder for production.

Sintered magnets are advantageous only in small sizes ranging from a fraction of a gram up to, say, 60 gms each. In a single die press 1,000-2,500 magnets can be pressed in an hour and for extremely large orders, multiple die presses with as many as 24 die cavities may be used. Some of the sintered alloys exhibit better magnetic properties than the corresponding cast material, and in cases where the requirements are extremely severe large sintered magnets may be made.

Rectangular magnets are pressed in the direction of the smallest dimension which should be at least 0.060 in. Cylindrical magnets are pressed axially and small magnets of this type should have a length not greater than three times the diameter. Cored holes may be inserted in the direction of pressing with a minimum diameter of 0.080 in. Where magnets are to be pressed on shafts it is not always possible to hold the cored-hole dimensions close enough

for a pressed fit. In such cases the hole is cored oversize and a mild steel insert placed in it before sintering. The shrinkage securely binds the insert in place on sintering and the hole may then be drilled and reamed to the correct size. During sintering the shrinkage amounts to several per cent. in all directions and due allowance has to be made in designing the dies. The tolerance maintained on sintered Alnico magnets varies from  $\pm 0.005$  in. on a dimension up to 0.125 in. to  $\pm 0.031$  in. on a dimension of 1.25-3.00 in.

## Silicon Bronze Welding Problems Overcome Through Inert-Arc Method

By PETER J. GURKLI

DIFFICULTIES have been encountered in welding a silicon bronze containing 3% Si and 1% Mn by the carbon arc method, due to porosity arising from the moisture in the atmosphere. A series of weld tests was made using the following processes: (1) Carbon arc; (2) Shielded metallic arc; (3) Helium-shielded arc; (4) Argon-shielded arc; and (5) Nitrogen-shielded arc. The welds were radiographed, tensile tested and bend tested (guided, free and nick-bends).

The carbon arc welds were excessively porous and could not be relied upon to pass the bend test whilst the nitrogen arc welds failed on the free bend test. Apart from this, difficulties were encountered with the nitrogen arc process because the nitrogen was unable to prevent burning away of the tungsten electrodes. As a result the manipulation was difficult and the weld contained a uniform dispersion of tungsten. The remaining processes produced quite satisfactory welds and although argon and helium are expensive, their use only brings the cost up to that of oxy-acetylene welding.

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